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FUSION TECHNOLOGY STUDIES
PROGRESS REPORT FOR PERIOD ENDING
DECEMBER 31, 1972

MASTER

OAK RIDGE NATIONAL LABORATORY

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ORNL-TM-4156

Contract No. W-7405-eng-26

METALS AND CERAMICS DIVISION

FUSION TECHNOLOGY STUDIES PROGRESS REPORT
FOR PERIOD ENDING DECEMBER 31, 1972

Compiled by

C. J. McHargue

MAY 1973

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OAK RIDGE NATIONAL LABORATORY
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CONTENTS

	<u>Page</u>
SUMMARY.	v
INTRODUCTION	vii
1. NEUTRON DAMAGE	1
1.1 Irradiation Experiments	1
1.1.1 Helium Injections.	1
1.1.2 Irradiation Experiments in HFIR.	3
1.1.3 Irradiation Experiments in EBR-II.	13
1.1.4 Irradiation Experiments in ORR	15
1.2 Helium Effects in CTR Materials	16
1.2.1 General.	16
1.2.2 Experimental Details	18
1.2.3 Results and Discussion	19
1.3 Mechanical Properties of Irradiated Molybdenum.	27
1.3.1 General.	27
1.3.2 Experimental	28
1.3.3 Results and Discussion	31
1.4 Studies of Swelling in Reactor Irradiated Metals.	48
1.4.1 General.	48
1.4.2 Experimental	49
1.4.3 Results and Discussion	49
1.4.4 Relationship of Swelling Results to CTR Operation.	59

	<u>Page</u>
2. DAMAGE SIMULATION STUDIES.	63
2.1 The Temperature Dependence of Fission Reactor Induced Swelling in Aluminum, Nickel, and Vanadium.	68
2.2 ORR-227, Temperature Dependent Neutron Damage Study	69
2.3 The Ion Bombardment Chamber	71
2.3.1 Design	71
2.3.2 Status of the Ion Bombardment Chamber on December 31, 1972.	79
2.3.3 Initial Experiments to be Performed.	79
2.4 Some Effects of Neutronically Generated Gases in Metals . .	80
 3. CHEMICAL COMPATIBILITY OF POTENTIAL BLANKET AND FIRST WALL MATERIALS	 86
3.1 Examination of the Forced-Circulation Lithium Loop.	86
3.1.1 Test Results	89
3.1.2 Significance of Results to CTR Program	93

SUMMARY

Tensile tests on EBR-II irradiated Nb, Ta, V, and their alloys showed elongations to failure in the range 4 to 17% for tests at 25, 400, and 650°C. Irradiation temperatures ranged from 390 to 700°C and fluences from 1.5 to 3.7×10^{22} neutrons/cm² (> 0.1 MeV). This result is in marked contrast to the Mo and Mo-0.5% Ti specimens which were brittle (zero elongation) to 550°C under similar irradiation and test conditions.

Creep tests of Nb and Nb-1% Zr containing 0, 2, or 18 ppm helium from alpha-injection have been run at temperatures between 800 and 1400°C. Ductility values as low as one-half that of the control samples were found.

Construction of experiment HFIR-CTR No. 2 was completed in September 1972, and irradiation between September 1972 and January 1973. Irradiation of experiment HFIR-CTR No. 1, inserted in the reactor in October 1971, is continuing.

Experiments HFIR-SS Nos. 2,3 were disassembled to recover vanadium alloy specimens for swelling studies and stainless steel specimens for the evaluation of the effects of very high helium contents.

The temperature dependence of irradiation swelling was determined for the pure metals aluminum (65 to 200°C), nickel (300 to 750°C), and vanadium (350 to 900°C). Voids were detected in vanadium at temperatures lower than expected and an ordered dislocation loop structure was observed in nickel at the lower temperatures.

The ion bombardment chamber was designed, constructed, and installed in the 6-MeV Van de Graaff.

Techniques for determining the distribution of gases injected into foils were examined in order to test the calculations on ion ranges and damage.

Assistance was provided to the Division of Controlled Thermonuclear Research, USAEC, in the assessment of potential materials compatibility problems in thermonuclear reactors. A critical review of the extant corrosion data for liquid lithium was completed and used to formulate corrosion requirements for qualifying lithium as a Controlled Thermonuclear Reactor (CTR) blanket material.

A 2 1/4 Cr-1 Mo-Nb stabilized steel loop was placed in operation with lithium early in FY 1973 and will continue operation through FY 1974. The pattern of interstitial impurity (C,N,O) migration between vanadium-titanium alloys and lithium was evaluated.

INTRODUCTION

Prior to 1972 there was a relatively small effort in the Metals and Ceramics Division which supported the studies of the Thermonuclear Division. In early 1972, the work was markedly increased as a portion of the Fusion Reactor Technology Program. Studies on several facets of radiation damage and the compatibility of lithium with refractory metal alloys are described in this report.

The radiation effects program consists of both neutron irradiation (supported by the Division of Controlled Thermonuclear Research) and ion bombardment simulation of damage (supported by the Division of Physical Research). The latter portion is being carried out jointly in the Metals and Ceramics and the Solid State Divisions. The studies conducted in the Solid State Division will not be covered in this report.

Fortunately, we had available many specimens from former or existing programs which could supply samples for studies in the fusion reactor program. Many of the results reported for the neutron damage and compatibility sections were obtained on such specimens.

Structural materials in fusion reactors will be subjected to radiation damage at least as severe as that encountered in advanced fission reactors. In these reactors the flux of high energy neutrons should generate vacancies and interstitials at rates which approach those in fast breeder reactors. Because of the higher energy of fusion reactor neutrons, transmutation products will be produced at rates far in excess of those in fission reactors.

Since present test reactors have fluxes of fast neutrons similar to those expected for thermonuclear reactors but with a softer energy spectrum, damage to structural materials in the former only approximates that to be expected in reactors of the latter type. It is expected that different point defect distributions may result from the different energy spectra and there will be lower rates of production of transmutation elements. There are no sources of 14 MeV neutrons that have sufficient fluxes for a materials testing and development program. We propose to simulate neutron radiation damage by ion bombardment in accelerators. Although such irradiations do not produce the same defect

distribution as will be caused by thermonuclear neutrons (because of the high energies required for enough penetration into the specimens by heavy ions), the use of accelerator irradiation does allow the introduction of transmutation products at the same rate relative to the number of displacements as will be encountered in service. Displacements equivalent to those caused by very high fast neutron fluences can be achieved in hours rather than the one or two years required in fast test reactors.

We are combining fission reactor irradiation experiments with simulation of damage by ion bombardment. In this way we expect to bridge the gap between the two methods and allow extrapolation to the conditions characteristic of fusion reactors.

Once the type and distribution of radiation damage are understood, material parameters such as composition and microstructure can be altered to produce structural materials that will resist or minimize any deleterious effects of the irradiation.

Neutron Irradiation of Potential First Wall CTR Materials

This task will study the irradiation damage produced by fission reactor irradiation of body-centered cubic metals and alloys, including alloys of niobium, vanadium, and molybdenum. These are the alloy systems most likely to satisfy the property requirements for a CTR wall. A major effort will also be devoted to evaluating the effects of transmutation products, especially helium, on irradiation damage of these metals. In-core positions in the High Flux Isotope Reactor (HFIR) and the Experimental Breeder Reactor (EBR-II) will be used. The reactor-irradiated specimens will be used to study mechanical properties effects and swelling and will provide standards against which such simulation techniques as ion bombardment can be compared and calibrated.

Ion Bombardment Simulation of Neutron Damage

The objectives of this task can be summarized as: (a) Establishment of a quantitative correlation between neutron damage and accelerator heavy ion damage so that extrapolations into the energy range of thermonuclear reactors can be made; (b) Definition of the nature and severity of problems due to radiation damage in materials which may be used as structural components in fusion reactors; and (c) Use of the technique

in conjunction with reactor irradiations to investigate material parameters in order to design a material to have the maximum resistance to deleterious effects of irradiation.

Compatibility Studies

Another major problem area concerns possible reactions between the tritium breeding blanket and the plasma containment vessel. Other compatibility problems are associated with convective heat transfer systems used to derive energy from the reactor.

Because of its high tritium breeding ratio and excellent heat transfer properties, liquid lithium affords an attractive dual-purpose blanket-coolant material for CTR applications. We are conducting corrosion studies with lithium to elucidate problems that are peculiar to the CTR concept.

This year we examined a forced-convection loop in which lithium had circulated for 3000 hr at a maximum temperature of 1370°C. Results of this loop test have been applied to predictions of thermal-gradient mass transfer under CTR conditions.

21,336
1. NEUTRON DAMAGE

F. W. Wiffen

1.1 Irradiation Experiments

1.1.1 Helium Injections

One of the critically important problems is the determination of the effects of helium (produced by n, α transmutations) on the mechanical properties, forms of displacement damage, and dimensional stability of structural materials. One way to simulate this transmutation effect is by injecting a uniform concentration of helium in metal samples. The injection is accomplished by stopping a variable-energy α -beam in the sample target. This technique, developed at ORNL by R. T. King, is discussed in detail in a report.¹ Using this technique, a sample rack containing 44 specimens each with a gage area of 0.1875 in. $\times$ 0.75 in. and with thickness in the range 0.010 to 0.011 in. is exposed in each run. A typical sample rack with 44 mounted samples is shown in Fig. 1.1. A beam current of 3.5 μ a (measured at the sample plane) produces a helium concentration of approximately 0.25 ppm (atomic) per hour of injection time.

Calculations of α -particle penetration depths, required degrader thickness, exact energy required in each run, and the approximate helium injection rates were made from the data for total stopping power of

¹R. T. King, Cyclotron Simulation of Neutron-Transmutation Produced Gases in Reactor Cladding and Structural Materials, ORNL-TM-3299 (April 1971).

Fig. 1.1. Typical Sample Rack, with 44 Mounted Specimens, Ready for ORIC Helium Injection. Tensile specimens are mounted with only the gage section exposed to the beam. The eight specimens showing through "windows" in the frame are for microscopy experiments. Note three thermocouples on the end sample.

α -beam in the tables from Northcliffe and Schilling.² For metals not given in their tables, the data were interpolated as a function of atomic number between the bracketing data pair given. Exact values of helium concentrations achieved in each injection run were determined by analysis by H. Farrar at Atomics International.

A listing of all specimens that have been helium injected for use in the CTR irradiation damage program is given in Table 1.1. Helium contents shown for runs 1 and 2 are measured values. The values shown for the other runs were determined by ratioing the appropriate experimental parameters against those for runs 1 and 2.

1.1.2 Irradiation Experiments in HFIR

The high fission neutron flux in HFIR makes it a highly desirable irradiation facility for use in the CTR program. Figure 1.2 compares the integral neutron spectrum of HFIR with the spectrum of EBR-II (the only fast fission reactor in the United States now available for radiation damage programs) and the calculated³ spectrum for CTR's of two different power densities. This plot shows that for neutrons with energy above 0.1 MeV (a commonly used cutoff in radiation damage fluence specifications), HFIR has the same flux as a fusion reactor with a 14-MeV neutron wall loading near 5 MW/m². The effectiveness of HFIR in producing displacement damage is more nearly equivalent to a CTR with a wall loading

²L. C. Northcliffe and R. F. Schilling, Nuclear Data Tables A 7, 223 (1970).

³Calculated CTR neutron spectrum at first wall supplied by Don Steiner, Oak Ridge National Laboratory.

Table 1.1. Helium Injection into CTR Materials Using ORIC Alpha-Beam

Run	Samples			Approximate Helium Content (ppm)	Intended Use
	Type ^a	Composition	Number		
CTR-1	0.25-in. gage	Nb	6	18	Ductility and Void Formation
		Nb-1% Zr	13	18	
CTR-2	0.25-in. gage	Nb	6	1.7	Ductility and Void Formation
		Nb-1% Zr	13	1.7	
CTR-3	0.1875-in. gage	Nb	5	1.3	Reactor Irradiation and Ductility
		Nb-1% Zr	13	1.3	
		Mo	11	1.3	
		TZM	4	1.3	
		V-20% Ti	4	1.3	
		Vanstar-7	6	1.3	
CTR-4	0.1875-in. gage	Nb	5	16	Reactor Irradiation and Ductility
		Nb-1% Zr	13	16	
		Mo	11	16	
		TZM	4	16	
		V-20% Ti	5	16	
		Vanstar-7	6		
CTR-5	0.1875-in. strip	Mo	1	< 0.1	Control Samples
		TZM	1	< 0.1	
CTR-6	0.1875-in. strip	Mo	1	5	Void Formation
		TZM	1	5	

^aGage section width is shown. Length of the gage portion of the sample is 0.75 in.

ORNL-DWG 72-11611

Fig. 1.2. Integral Flux in HFIR and Two Positions of EBR-II Compared to the Calculated Flux for CTR at Two Power Densities.

nearer 1 to 2 MW/m² because of the lower energies of fission neutrons compared to fusion reaction neutrons. Transmutation reactions cannot be as well simulated in HFIR as can the displacement damage because of the strong energy dependence of most of the transmutation reactions. One exception to this statement is the case of the two-step reaction resulting in the production of helium from nickel⁴ in the HFIR spectrum, and this will be useful to our program in producing high concentrations of helium in austenitic stainless steels.

It should be noted that although Fig. 1.2 shows the center core of EBR-II with a higher neutron flux than HFIR, the spectrum in HFIR is harder and the integral flux for the two reactors is equal for fluxes above 0.6 MeV. The HFIR has a further advantage in operating with a plant factor > 90% while the EBR-II plant factor is about 60%. Under these conditions, the fluence per year of neutrons with energy > 0.1 MeV is very nearly equal for the two facilities.

Irradiation experiments in the HFIR peripheral target positions are not instrumented. Neither thermocouples nor heaters can be used. Experiments to run at temperatures above reactor ambient (~ 55°C) utilize the very high nuclear heating in the HFIR core, about 35 W/g in niobium. In a typical HFIR experimental position, shown schematically in Fig. 1.3, a specimen or a susceptor tube containing specimens is centered in the containment tube by thin centering disks. The components of a specimen position containing a rod tensile specimen, similar to the lower position shown schematically in Fig. 1.3, are shown in Fig. 1.4. The

⁴W. N. McElroy and H. Farrar IV, pp. 187-229 in Radiation-Induced Voids in Metals, USAEC Symposium Ser. 26, CONF-710601 (April 1972).

ORNL-DWG 69-8936

Fig. 1.3. Schematic Representation of Two Sample Positions in a Typical HFIR Irradiation Experiment. The upper position is shown containing a SiC passive temperature monitor. The same geometry is used to hold a stack of microscopy disks. The lower position contains a rod mechanical properties specimen.

Fig. 1.4. Components of a Specimen Holder Assembly and Rod Mechanical Properties Specimen for a HFIR Irradiation Experiment. When assembled, this will occupy one of fifteen positions in a typical HFIR experiment.

components of a specimen position in which a series of electron microscope specimens will be irradiated in a containing susceptor tube are shown in Fig. 1.5. The heat produced by gamma heating in the specimen is conducted across the helium-filled gas gap to the specimen holder. Control of the thickness of this conduction gap sets the temperature gradient between the specimen and specimen holder and thus sets the specimen irradiation temperature. Temperatures in the CTR experiments have been designed in the range 375 to 950°C. One HFIR experiment contains 15 of these experimental positions (although two may be used to accommodate longer specimens).

Two CTR experiments are currently in the HFIR; HFIR-CTR-1 was inserted in October 1971 and now has an accumulated fluence near 5×10^{22} neutrons/cm² (> 0.1 MeV), and HFIR-CTR-2 went into the reactor September 1972. This experiment will be removed in January 1973 with a midplane fluence near 1.4×10^{22} neutrons/cm² (> 0.1 MeV). A removal date has not yet been set for HFIR-CTR-1.

HFIR-CTR-1 is a standard, 15-position design containing one passive temperature monitor, two sets of microscopy disks, and 12-rod tensile specimens. Temperatures range from 450 to 950°C, and the detailed specimen distribution as a function of irradiation temperature is given in Table 1.2. The experiment is designed to yield results on the influence of irradiation temperature and helium content on the microstructural changes, especially swelling, produced by neutron irradiation of Nb and Nb-1% Zr. The experiment will also test the validity of using ¹⁰B-doped samples [where helium is produced by ¹⁰B(n,α)⁷Li] as a method of simulating (n,α) transmutation reactions. Tensile tests on the rod

Fig. 1.5. Components of a Specimen Holder Assembly of the Type Used in Irradiating a Stack of Microscopy Disk Specimens.

Table 1.2. Specimen Distribution in Irradiation
Experiment HFIR-CTR-1

Alloy ^a	Irradiation Temperature, °C				
	450	550	650	800	950
Nb				5D	
Nb + He			5D	5D	
Nb + B		M ^b	M + 5D	5D	
Nb-1% Zr			M + 10D	10D	
Nb-1% Zr + He			5D	10D	
Nb-1% Zr + B	2M	M	4M + 15D	M + 13D	M
Type 316 SS			5D	3D	

^aAlloy designation (+He) represents α -injected material.
Designation (+B) represents samples doped with ^{10}B during melting.

^bM = rod-type mechanical property specimen. D = disk for transmission electron microscopy.

specimens will be conducted to obtain some information on the ductility of irradiated Nb-1% Zr.

HFIR-CTR-2 is a similar experiment designed to provide microscopy specimens of the same materials as in the first experiment but for more irradiation temperatures and for a lower neutron fluence. Pressurized tube specimens of type 316 stainless steel and Nb-1% Zr are also included to evaluate the feasibility of using the HFIR facility to examine the effect of stress on samples during irradiation. The experiment also includes two rod specimens for tensile ductility tests. The detailed distribution of specimens in this experiment is shown in Table 1.3.

Table 1.3. Specimen Distribution in Irradiation
Experiment HFIR-CTR-2

Alloy ^a	Irradiation Temperature, °C					
	375	450	500	550	650	800
Nb		5D ^b		5D	5D	5D
Nb + He				5D	2D	5D
Nb + B				5D	5D	5D
Nb-1% Zr		10D	2T	10D	2M + 10D	10D
Nb-1% Zr + He		10D			5D	5D
Nb-1% Zr + B		10D		5D	10D	10D
Mo		5D		5D	8D	5D
V-20% Ti				5D		
Vanstar-7		6D		5D		5D
Type 316 SS	2T	5D		5D	5D	

^aAlloy designation (+H) represents α -irradiated material. Designation (+B) represents sample doped with ^{10}B during melting.

^bD = disk for transmission electron microscopy. T = pressurized tube specimen. M = rod-type mechanical property specimen.

1.1.3 Irradiation Experiments in EBR-II

The EBR-II reactor is currently the best irradiation facility for high fluence neutron damage experiments. Although the flux level is not very much greater than HFIR, the volume available for experimental irradiations is much larger. Like HFIR, EBR-II experiments are generally uninstrumented and irradiation temperatures are determined by the same general method as in HFIR.

Oak Ridge National Laboratory experiments designed and built for EBR-II irradiations as parts of other programs have specimens fitted into specimen holders to produce a nearly solid cylindrical holder assembly. A typical loaded specimen holder is shown schematically in Fig. 1.6. Desired irradiation temperatures are achieved by varying the diameter of the specimen holder and, thus, the width of the gas-filled heat-conduction gap between the holder and the experimental containment tube.

Although Fig. 1.6 shows only button-head type rod tensile specimens, several other specimen types have been included in similar specimen holders. A detailed description of the design, fabrication, and assembly of a complete ORNL experimental subassembly for EBR-II irradiations is given by Bloom et al.⁵

Most of the high-fluence irradiated refractory-metal specimens reported on here were irradiated either in experimental subassembly X034 (and X034-A), irradiated in row 2 of EBR-II or in experimental subassembly X035, irradiated in row 7 of the reactor. Irradiation temperatures range

⁵E. E. Bloom, J. W. Woods, and A. F. Zulliger, Description of EBR-II Subassembly X-100, ORNL-TM-3389 (June 1971).

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Fig. 1.6. Loaded Specimen Holder of the Type Used in EBR-II Materials Irradiation Experiments.

from 400 to 1000°C and fluences on the various refractory-metal samples ranged from 1.5 to 4.5×10^{22} neutrons/cm² (> 0.1 MeV). The irradiation parameters are given with the presentation of results on these samples. These specimens were irradiated as part of other programs as indicated in the discussion.

1.1.4 Irradiation Experiments in ORR

Irradiation in the Oak Ridge Research Reactor (ORR) offers the advantages of ample volume for a large number of specimens and the ability to instrument experiments in order to provide positive temperature control by the use of furnaces surrounding specimens. One such experiment, ORR-227, was run in the B8 position of the reactor core. The primary purpose of the experiment was to provide swelling vs irradiation temperature data in support of the CTR ion damage program, and those features of the experiment and the experimental design are discussed in Part 2 of this report. The large volume in this experiment allowed us to include several refractory-metal specimens for use in the neutron irradiation program. This experiment provides specimens for low fluence data in the determination of the effect of fluence and irradiation temperature on the microstructural response to neutron irradiation. Specimens were chosen from the same materials used in other experiments. Typical specimens were rods $1/8$ in. in diameter and 0.67 in. long. To protect these refractory metals from an oxidizing atmosphere, the rods were sealed in close-fitting stainless steel capsules with a helium fill gas. The capsules were then mounted in the wire-wound furnaces, thermocouples were attached, and during irradiation the capsules were held at various temperatures in the range 400 to 900°C. The distribution of specimens as a function of irradiation temperature is shown in Table 1.4.

Table 1.4. Refractory Metal Sample Distribution by Irradiation
Temperatures in ORR-227 Irradiation Experiment.

Fluence in Experiment Approximately
 1.0×10^{21} neutrons/cm² (> 0.1 MeV)

Alloy ^a	Irradiation Temperature, °C							
	400	500	600	700	750	800	850	900
Nb	1	1	0	1	0	1	3	1
Nb-1% Zr	1	1	0	1	0	1	2	1
Nb-1% Zr with He ^b	0	0	0	0	0	5D	5D	0
Nb-5% Zr	0	1	0	0	1	0	2	0
Mo	0	1	1	1	1	1	2	1
TZM	0	0	1	1	0	1	1	1

^aSamples unmarked are 0.123-in.-diam rods approximately 0.67 in. long. Samples identified as "D" are disks punched from 0.010-in.-thick sheet material.

^bSamples containing 1.7 and 18 ppm He are included in each set of five disks.

Irradiation of this experiment is complete and the experiment has been disassembled. The refractory-metals specimens are now in hot-cell storage awaiting examination.

21,337

1.2 Helium Effects in CTR Materials

1.2.1 General

As helium produced in (n,α) reactions is believed to be the most important of the transmutation products of 14-MeV neutron irradiation, we have singled it out for separate study in our program. Helium has two important effects on metals. First, it affects the failure mode during

elevated-temperature loading and results in reduced ductility. This helium embrittlement has been investigated in considerable detail in face-centered cubic metals, especially in austenitic stainless steels and nickel-base alloys but has received little attention in bcc metals. Detrimental effects of helium on tensile ductilities have been demonstrated in vanadium-base alloys^{6,7} and in one niobium alloy.⁷ There have been no published results of creep tests on these materials. The second major effect of helium is on the irradiation-produced swelling that results from cavity formation. The agglomeration of helium into equilibrium bubbles can lead to swelling, and the formalism of this problem has been reviewed in detail by Martin.⁸ Although Martin's treatment predicts appreciable swelling under certain conditions, Wiffen⁹ has argued that most of the helium produced will be captured in void-like cavities (with gas pressures well below equilibrium values). The high concentration of these void-capture sites that form in irradiated bcc metals should not allow the formation of the fewer, larger helium bubbles that are required for appreciable swelling due to helium. The latter process will occur if the pressure in the bubble exceeds that

⁶K. Ehrlich and H. Bohm, Radiation Damage in Reactor Materials, vol. II, IAEA, Vienna (1969), pp. 349-355.

⁷D. Kramer, Fusion Reactor First Wall Materials, WASH-1206 (1972), pp. 50-51.

⁸D. G. Martin, Radiation Damage Effects in the Containment Vessel of a Thermonuclear Reactor, CLM-R-103 (March 1970).

⁹F. W. Wiffen, Proc. Inst. Working Sessions on Fusion Reactor Technology, ORNL, June 28-July 2, 1971, CONF-710624, pp. 140-174.

balanced by the surface tension. For example, in stainless steel,¹⁰ the helium from (n,α) reactions during CTR service will probably cause the nucleation of more cavities than would be produced during irradiation in a fission reactor.

Experimental results on the effects of helium on void formation and swelling processes will be discussed in Section 1.4. The two irradiation experiments HFIR-CTR-1 and -2 include Nb and Nb-1% Zr which contain helium produced both by α -injection and by the reaction $^{10}\text{B}(n,\alpha)^7\text{Li}$.

The remainder of this chapter is devoted to the examination of the effect of helium at concentrations near 2 and near 20 ppm (atomic) on the creep test ductility of Nb and Nb-1% Zr.

1.2.2 Experimental Details

The material used for specimens in evaluation of the effects of helium content on the creep ductility was chosen from typical material from commercial suppliers. The zirconium content of the niobium was 0.03%; in the Nb-1% Zr, it was 0.9%. Interstitial impurity content in each was < 400 ppm (C + N + O). Both materials were cold rolled from a thickness of 0.06 to 0.010 in. Tensile specimens having a length of 2.75 in. and a reduced gage length section 0.25 in. wide by 0.75 in. long were machined from the sheet. The cleaned specimens were annealed in a vacuum at pressures $\leq 1 \times 10^{-6}$ torr. The niobium samples were annealed 1 hr at 1150°C, the Nb-1% Zr 1 hr at 1200°C.

Helium injection in these specimens was accomplished in runs ORIC-CTR-1 and -2. The helium concentration achieved in run 1 was 18 ppm (atomic) and in run 2 was 1.7 ppm.

¹⁰E. E. Bloom and J. O. Stiegler, J. Nucl. Mater. 36, 331 (1970).

The creep tests were conducted in a cold-wall vacuum apparatus in which the specimen was heated by a tantalum wire furnace. The testing described here was generally done at pressures $\leq 2 \times 10^{-7}$ torr. Test temperatures were controlled and monitored by Pt vs Pt-10% Rh thermocouples wired to the gage length portion of the sample. The temperature gradient along the gage length was $< 5^{\circ}\text{C}$.

The specimens were stressed via a dead-weight loading system external to the vacuum furnace. The load is transmitted to the specimen pull-rod on a direct-ratio pulley system. The pull-rod passes out of the furnace through a conventional U-cup vacuum seal. The strain was measured at the pull-rod with a dial indicator. The creep ductility values reported reflect plastic deformation only.

Scanning electron microscopy has been used to examine fracture surfaces and to look for helium effects on the fracture mode.

1.2.3 Results and Discussion

While the control specimens displayed the expected behavior, with failure within the gage section, the doped specimens all failed at the end of the gage section with the failure approximately at the dividing line between the injected and uninjected parts of the specimen. The creep-rupture times and fracture elongation for tests on niobium at 1000°C are given in Table 1.5. In the first series of tests at 4000 psi, the rupture times for specimens with 1.7 and 18 ppm He were both longer than the control tests by approximately a factor of 4. The elongation for the specimen containing 1.7 ppm He was only slightly less than that of the control sample, but the specimen containing 18 ppm He showed only one-third the ductility of the control sample. Examination of the

Table 1.5. Creep-Rupture Properties of Nb and Nb-1% Zr With and Without Helium

Alloy	Temperature (°C)	Stress (10 ³ psi)	Helium Content					
			No Helium		1.7 ppm		18 ppm	
			Rupture Life (hr)	Elongation (%)	Rupture Life (hr)	Elongation (%)	Rupture Life (hr)	Elongation (%)
Nb	1000	6	3.9	54.3				
Nb	1000	4	64.0	46.5	277.1	42.4 ^a	208.6	18.1 ^a
Nb	1000	4	72.0	58.7			36.9 ^a	12.9 ^a
Nb-1% Zr	1000	10	781.4	33.8				
Nb-1% Zr	1000	14	32.9	33.9	61.5	14.9	61.3	16.9
Nb-1% Zr	1000	21	1.0	26.5				
Nb-1% Zr	1200	4	455.7	35.7				
Nb-1% Zr	1200	5	242.4	62.6				
Nb-1% Zr	1200	6	77.0	62.2	53.2	46.5	65.3	46.2
Nb-1% Zr	1200	7	37.6	67.0				
Nb-1% Zr	1200	8	7.3	66.0				
Nb-1% Zr	1200	9	2.9	60.0			3.1	51.5
Nb-1% Zr	1400	5	580.2	85.6			296.6	97.1

^aIn all helium-containing niobium specimens, the failure occurred at the end of the helium-doped gage section.

specimens, however, showed the failure at the end of the gage section, with very little evidence of deformation in the helium-injected material. In one case, "necking" had also occurred at the opposite end of the gage section to the fracture.

Failures of all of these samples occurred after the samples had necked down to a knife-edge. This near 100% reduction in area to fracture indicates little or no effect of the helium on ductility of niobium at 1000°C when reduction in area is used as the measure of ductility. The reduction in elongation to fracture in these samples then represents a reduced "effective" gage length over which the deformation occurred in the helium-doped samples, rather than any effect on the fracture mode. Detailed fractography to confirm the lack of effect on the failure mode has not yet been completed.

Creep-rupture data for Nb-1% Zr samples tested at 1000, 1200, and 1400°C are included in Table 1.5. The results show less helium effect on the creep ductility than would have been expected based on tensile results on vanadium alloys.⁶ The data at 1000°C show only a modest dependence of ductility of the control tests on stress, with the ductility in a 1-hr test three-quarters that in 30- and 800-hr tests. Helium-containing samples with 1.7 and 18 ppm He tested at 1000°C and 14,000 psi had rupture lives approximately twice that of the control tests and rupture elongations approximately one-half that of the control values. The creep curves for the three tests at 1000°C and a stress of 14,000 psi are shown in Fig. 1.7. All three tests show the three-stage creep behavior. The lower ductility in the helium-doped samples is reflected in a reduced third-stage creep. Scanning electron microscope fractography on the same three samples did not

Fig. 1.7. Elongation Vs Time Curves for Creep Tests of Nb-1% Zr at 1000°C at a Stress of 14,000 psi. Curves are shown for a control sample and samples with 1.7 and 18 ppm He. Note different scales on abscissa.

reveal any differences in the fracture mode. Typical areas on the three fractures are shown in Fig. 1.8. Fracture in these samples has resulted from the metal necking down to a chisel-point fracture edge that extends across the width of the sample. There are a few dimples on the fracture edges, corresponding to the opening of the voids that form in the necked zone during deformation. Reduction in area for these samples is essentially 100%.

Creep tests at 1200°C showed a generally greater ductility than tests at 1000°C, with fracture elongations approximately twice those at the lower temperatures. At this temperature, the lowest elongation was recorded in the lowest stress-longest time test (36% elongation in a 456-hr test), while other tests lasting from 3 to 250 hr at various stresses had elongations of $64 \pm 4\%$. In tests at 1200°C and 6000 psi, helium-injected samples had slightly shorter rupture lives and approximately three-quarters of the rupture elongation of the control sample. Creep curves of a control sample and samples with the two helium levels are shown in Fig. 1.9. (The broken curve for the 18 ppm He sample shows the recorded data. It is believed that this irregularity was due to a slight temperature excursion during the test.) In these curves, too, the general form of the curves is the same for both control and helium-doped samples. The reduced ductility in the helium-doped samples has resulted from the reduced amount of third-stage creep. Fractography on these samples did not reveal any difference in failure mode due to the helium doping. In all three samples, failure had been preceded by necking to almost 100% reduction in area. The final fracture in all three samples left a chisel-point fracture, with some ductile dimples along the failure edge.

PHOTO 4019-72

NO HELIUM

1.7 ppm HELIUM

18 ppm HELIUM

Fig. 1.8. Fracture Surfaces of the Specimens from Tests Shown in Fig. 8.

Fig. 1.9. Elongation Vs Time Curves for Creep Tests of Nb-1% Zr at 1200°C at a Stress of 6000 psi. Curves are shown for helium contents of 0, 1.7, and 18 ppm.

A test at a higher stress, and thus shorter creep-rupture time, showed less effect of the 18 ppm He than found at shorter creep times. In this test at 1200°C and 9000 psi, the creep-rupture times were comparable for control and helium-containing samples, with the helium-injected sample having 85% of the rupture elongation of the control sample.

Tests conducted at 1400°C at a stress of 5000 psi showed 13% more elongation to rupture in the 18 ppm He-doped specimen than in the control. However, there probably was considerable grain growth during these tests and more detailed specimen examination is still required.

In niobium tested at 1000°C, ductilities as low as one-quarter of the control value have been observed in helium-injected specimens. However, the reduction of ductility seems to be due to a reduced "effective" gage length, with deformation occurring at the end of the gage section outside of the helium-injected zone. The ductility reduction in these samples cannot yet be accepted as a proven helium effect. In Nb-1% Zr ductility, reductions were found in creep-rupture elongations in tests at 1000 and 1200°C, but not in a test at 1400°C. The mechanism of the reduction in elongation is not clear, as there was no difference observable in the fracture modes. All samples examined showing chisel-edge fractures with near 100% reduction in area and with a few ductile-mode dimples observed on the fracture edge. The tests at 1400°C probably were complicated by grain growth during the test. In no case did we observe loss of ductility as great as the factor of ten reduction in tensile ductility produced in vanadium alloys by the injection of 1 ppm He.

1.3 Mechanical Properties of Irradiated Molybdenum

1.3.1 General

Molybdenum or molybdenum alloys are attractive candidate first wall materials and their use has been suggested in several conceptual designs. These metals have high strength at operating temperatures and good compatibility with lithium, molten salt or helium coolants. The fabricability, especially the welding, of molybdenum, however, is very difficult. A significant unknown factor is the performance of molybdenum under neutron irradiation in fusion reactor service.

Some changes produced in the mechanical and physical properties of molybdenum by neutron irradiation have been determined. These experiments have generally involved irradiation at reactor ambient temperatures (100°C or lower) and fast fluences of 10^{20} neutrons/cm² or lower. Properties have been measured in the as-irradiated state and following postirradiation annealing.

The limited available data show that the most critical property of molybdenum alloys is the post-irradiation ductility in tests representative of some part of the CTR operating cycle. Data in the literature that bears on possible CTR use of molybdenum is incompletely documented. Tensile tests after irradiation at 490°C to a total fluence of 1.5×10^{22} neutrons/cm² show very low post-irradiation ductilities. These ductility data, presented in Table 1.6, show a completely brittle failure at 600°C and small ductility values at 700 and 800°C. These ductility data suggest that the irradiation has raised the ductile-to-brittle transition temperature (DBTT) to above 600°C. The strain rate for these tests was not cited, but the DBTT is very dependent on this parameter.

Table 1.6. Tensile Ductilities of Irradiated Molybdenum.^a
Irradiation at 490°C to 1.5×10^{22} neutrons/cm² (total).

Test Temp. (°C)	Total Elongation ^b (%)					
	Warm Worked ^c		17 μ m Grains		50 μ m Grains	
	Control	Irradiated	Control	Irradiated	Control	Irradiated
600	4.2	0	32.3	0	30.4	0
700	3.4	0.5	29.2	0.5	21.9	1.2
800	4.6	2.9	33.8	1.8	25.9	2.7

^aUnpublished Harwell data quoted in Reactor Materials 10, 41 (1967).

^bTest and material conditions are not available.

^cThis material condition is assumed, not stated. The control ductility values are typical of a warm worked or stress relieved state.

The work reported in this chapter was initiated by John Moteff (now at University of Cincinnati) while at GE-NSP; a program originally aimed at qualification of refractory metals for fission reactor service. Most of the tensile testing was completed in that phase of the program. The fractography, microscopy and data analyses have been extended under the CTR program because of the interest in molybdenum.

Specimens of Mo, Mo-0.5% Ti and Mo-50% Re irradiated in Row 2 of the EBR-II reactor have been tensile and creep tested. The results are reported here with emphasis on the post-irradiation ductility and the fracture mode of these alloys.

1.3.2 Experimental

The molybdenum and the Mo-0.5% Ti specimens were fabricated from arc-cast material. Standard buttonhead tensile specimens with a 1/8-in.-diam by 1 1/8-in.-long gage section were ground from rods. The

post-machining final anneal was in vacuum for 1 hr at 1350°C for the molybdenum and at 1450°C for the Mo-0.5% Ti. The Mo-50% Re was produced from sintered powder. Specimens machined from this material were given a final anneal in vacuum of 1 hr at 1400°C.

Irradiation was in subassembly XO34 in row 2 of EBR-II. The specimens were loaded in holders as shown in Fig. 1.10 and the irradiation temperatures were set as described in Section 1.3.1. All control results were obtained from specimens in the annealed conditions; there was no attempt to age specimens to duplicate the thermal history of the reactor irradiation.

Tensile tests were conducted on an Instron testing machine in a flowing helium environment. Specimens were held for a constant equilibration time of 30 min after reaching test temperature before the start of test. Stresses reported are engineering values and the elongations are taken from the crosshead travel of the testing machine. Creep tests were conducted in the same cold-wall vacuum test stands discussed in Section 1.2. In this case, the direct ratio pulley system was replaced by a 4:1 ratio lever to transmit the dead weight load to the pull-rod system. As before, the strain was measured at the pull rod with a dial indicator.

Scanning electron microscopy was used for evaluation of fracture surfaces, at magnification ranging from 50 to 10,000. The scanning microscopy in this section was done with the assistance of Robert K. Bennett, Jr., of the Union Carbide Corporation's Y-12 Plant. A limited amount of electron microscopy on these samples and samples irradiated under similar conditions characterized the as-irradiated microstructures, and was done with the assistance of C. Jones and J. O. Stiegler.

Fig. 1.10. Typical Examples Illustrating the Three Types of Tensile Behavior Found in Molybdenum Alloy Specimens. Curve A illustrates a fully brittle failure; curve B a ductile failure with no work hardening ability in the sample; and curve C the normal behavior of unirradiated specimens tested above the DBTT.

1.3.3 Results and Discussion

(a) Tensile Strength Properties.- Results of the tensile tests and the irradiation parameters for each set of samples are given in Table 1.7. In general the tensile results on the Mo and the Mo-0.5% Ti samples are similar and they will in most cases be treated as a single set of data. In these materials we observed three distinctly different types of tensile behavior. The three types are shown for tests on Mo-0.5% Ti samples at 400°C in Fig. 1.11. Control tests (curve C) gave the typical type of tensile curve. The curve shows a well-defined yield strength, the load gradually increasing above this value until the ultimate tensile strength is reached, and then dropping rapidly as the specimen deforms locally by necking until failure occurs. There were two types of results on irradiated samples. At lower test temperatures and higher strain rates the specimens were fully brittle. The load increased along the elastic modulus line to at least 100,000 psi and then failure occurred, with 0.2% or less deviation from the modulus line. There was no reduction in area accompanying the fracture and the first crack to form propagated to failure. This result is shown by curve A of Fig. 1.11. The third type of result, curve B, was found in irradiated samples tested either at slower strain rates or at higher temperatures. The maximum load was reached within 0.2% or less of the elastic modulus line and plastic instability occurred coincident with yielding. The specimen necked from this point to failure, with greater than 50% reduction in area and elongation of less than 10% (in a 1-in. gage length) before fracture.

Table 1.7. Tensile Properties of Molybdenum Alloys Irradiated at 425°C

Test Temp. (°C)	Strain Rate (min ⁻¹)	Yield Stress, psi		Ultimate Tensile Stress, psi		Uniform Elongation, %		Total Plastic Elongation, %	
		Unirra- diated (0.2% offset)	Irra- diated (maximum)	Unirra- diated	Irra- diated	Unirra- diated	Irra- diated	Unirra- diated	Irra- diated
A									
-196	0.02	131,000	--	131,000	--	0.1	--	0.1	--
20	0.02	57,900	--	72,000	--	22.0	--	54.6	--
400	0.02	12,600	113,300	36,300	113,300	27.3	0.1	39.4	0.1
550	0.02	10,500	101,000	33,500	101,000	31.7	0.2	41.8	0.2
550	0.0002	9,400	85,000	32,100	85,000	28.1	0.1	37.1	6.5
650	0.02	10,700	86,300	32,400	86,300	29.7	0.1	42.3	8.4
B									
20	0.02	52,700	--	80,500	--	21.0	--	59.9	--
400	0.02	14,400	118,000	44,500	118,000	34.0	0.1	50.1	0.1
400	0.002	15,000	102,600	41,700	102,600	32.9	0	47.5	0
400	0.0002	17,100	101,200	40,700	101,200	30.9	0.1	47.3	7.0
650	0.02	14,000	94,400	40,300	94,400	29.3	0.1	39.6	7.9
C									
400	0.02	63,300	144,300	87,400	144,300	20.8	0.1	31.7	0.1
650	0.02	45,000	147,300	76,100	147,300	12.5	0.2	20.3	0.2
800	0.02	41,900	141,400	68,000	141,400	12.1	0	21.1	0

^A Molybdenum [3.45×10^{22} neutrons/cm² (> 0.1 MeV)]

^B Mo-0.5% Ti [3.0×10^{22} neutrons/cm² (> 0.1 MeV)]

^C Mo-50% Re [1.5×10^{22} neutrons/cm² (> 0.1 MeV)]

Fig. 1.11. The Ultimate Tensile Strength of Irradiated and Control Samples of Mo and Mo-0.5% Ti. Irradiation at 425°C, the Mo to 3.45×10^{22} neutrons/cm² (> 0.1 MeV) and the Mo-0.5% Ti to 3.0×10^{22} neutrons/cm². Data points joined by solid lines are for tests conducted at a strain rate of 0.02/min. Data points at the end of broken lines tested at 0.0002/min.

In the temperature range 400 to 650°C the strength of the Mo and Mo-0.5% Ti control samples were only slightly temperature and strain rate sensitive. In the irradiated samples, for all test conditions, the ultimate strength was reached on the elastic modulus line; that is, the maximum load occurred at the yield point. The ultimate strength was dependent on both the temperature and strain rate for these samples.

Neutron irradiation of Mo and Mo-0.5% Ti under the conditions used here produces a complex microstructural damage. This damage strengthens the matrix metal, by increasing the resistance to dislocation motion, and causes an increase in the yield stress of as much as a factor of 10. In the annealed samples work hardening processes further strengthen the matrix until the ultimate strength is reached. In the irradiated samples the matrix does not work harden beyond the yield. As a result, the irradiation strengthening (ultimate strength) is a factor of 3 or less. The ultimate tensile strengths of the irradiated and control tests of Mo and Mo-0.5% Ti are compared in Fig. 1.12. Note that the stress producing brittle failure at -196°C in the control sample is slightly greater than the brittle failure strength in the irradiated samples at 400°C. A common interpretation of brittle failure in bcc metals, reviewed by Rosenfield et al.,¹¹ is that brittle failure occurs at temperatures for which the yield stress is greater than the cleavage stress. The cleavage stress is not strongly temperature or strain rate dependent in any given metal. The results available here may fit any one of three models: (1) The cleavage stress

¹¹A. R. Rosenfield, E. Votava, and G. T. Hahn, Ductility, American Society for Metals, Metals Park, Ohio (1968), pp. 63-85.

Fig. 1.12. Total Tensile Elongation as a Function of Test Temperature for Molybdenum. Irradiated specimens exposed at 425°C to a fluence of 3.45×10^{22} neutrons/cm² (> 0.1 MeV). The discontinuity at 550°C in the curve for irradiated samples is a strain rate effect.

may be weakly temperature dependent, dropping from 130,000 psi at -200°C to about 110,000 psi at 400°C and dropping slightly lower at 550°C .

In this model, there is no effect of the irradiation on the cleavage stress.

(2) The cleavage stress may be truly temperature independent and the cleavage stress is lowered by irradiation. (3) The cleavage stress is both temperature and irradiation-damage independent but, because of sensitivity to notches and other imperfections, observed values define a wide scatter band. The data available here can fit any of these models. A much larger collection of data that can be treated statistically will be required to determine if the neutron irradiation damage influences the brittle fracture (cleavage) stress.

The Mo-50% Re behaved differently from the other molybdenum samples. Tensile tests at 400, 650, and 800°C all showed brittle failure, with failure stresses essentially temperature independent at $144,300 \pm 3,000$ psi. The failure stress of the irradiated samples was from 1.6 to 2 times the ultimate stress of control samples tested at the equivalent temperatures. The ultimate stress of the control samples decreased by 30% over this temperature range 400 to 800°C .

(b) Tensile Ductility.— The tensile ductilities, given in Table 1.7, show a severe effect of neutron irradiation. The irradiated samples that failed in a brittle mode showed only elastic deformation, with 0 to 0.2% deviation from the modulus line. The irradiated samples that failed in a non-brittle mode showed tensile elongations of less than 10% in a 1-in. gage section, with all deformation restricted to the necking zone and thus less than 0.2% uniform elongation.

The elongation data for the tests on unalloyed molybdenum samples is plotted in Fig. 1.13. This figure shows that the DBTT is between

Fig. 1.13. Typical Areas on the Fracture Surfaces of Mo-0.5% Ti Tensile Specimens. Fractographs A, B, and C are taken from the specimens represented by tests A, B, and C in Fig. 1.10. Original SEM magnification 1000 $\times$.

-200 and 20°C. The DBTT for the irradiated molybdenum is about 550°C, but it is strongly strain rate dependent. At a tensile strain rate of 0.02/min our limited data show the DBTT to be above 400°C for Mo-0.5% Ti and above 550°C for Mo but below 650°C for both. As mentioned earlier, these two materials exhibit similar behavior and thus we assume the DBTT at this strain rate is between 550 and 650°C for both materials. Tests at lower strain rates showed that irradiated Mo-0.5% Ti remained brittle at 0.002/min but yielded and showed limited ductility at 0.0002/min at 400°C. The unalloyed Mo was tested at this low strain rate only at 550°C, in which test it was ductile. We conclude that the DBTT for the irradiated Mo and Mo-0.5% Ti is below 400°C at a strain rate of 0.0002/min. Unfortunately, we did not have enough specimens to bracket the DBTT at this strain rate.

The irradiated specimens that showed elongations greater than 0.2% at failure all exhibited load-elongation curves similar to curve B in Fig. 1.11. This type of tensile curve can be described as exhibiting "work softening," with the resistance to plastic deformation (in the deforming neck region) decreasing with deformation. This work softening may be the result of the deformation occurring by dislocation channelling (see, for example, Tucker et al.¹²) with the matrix of a deforming sample offering decreasing resistance to dislocation passage as channels are swept free of the irradiation-produced dislocation loops.

The irradiated specimens of Mo-50% Re were only tested at one strain rate (0.02/min) and failed in a brittle mode at 400, 650, and 800°C. This indicates a DBTT in excess of 800°C for this irradiated alloy.

¹²R. P. Tucker, M. S. Wechsler, and S. M. Ohr, J. Appl. Phys. 40, 400 (1969).

(c) Creep Properties.-- Two irradiated specimens of Mo and one of Mo-50% Re, as well as appropriate control samples, have been creep tested to evaluate the ductility at higher temperatures and lower strain rates than used in the tensile tests. The creep test parameters and results are given in Table 1.8.

The irradiated Mo-50% Re sample was from the same set of specimens as the specimens of this alloy that were tensile tested. At 800°C control samples elongated 21 and 22% to failure in tensile and 22 hr creep tests, respectively. At the same creep stress, 60,000 psi, the irradiated specimen showed no deformation after 1500 hr. Further step loading to increase the stress on the sample produced approximately 0.5% elongation in 290 hr at 80,000 psi and a further approximately 0.5% in 210 hr at 90,000 psi before fracture occurred. The total accumulated elongation to failure was 1.1% during a total test time of 2,119 hr.

The two irradiated molybdenum samples that have been creep tested were irradiated in EBR-II to a fluence of 6.1×10^{22} neutrons/cm² (> 0.1 MeV). The irradiation temperature was calculated after irradiation to be initially 860°C but rising to an end-of-life value of 1140°C. The irradiated sample tested at 1200°C at a stress of 8,000 psi gave a strain-time curve of the same general shape as the control sample tested under the same conditions. However, the elongation to fracture was only 80% of that of the control sample. The rupture life was 428 hr, compared to a control sample rupture life of 13.7 hr. This comparison shows a significant irradiation strengthening in the 1200°C creep test but only a small effect on the creep ductility. The one irradiated sample being creep tested has been under test for 5000 hr at 750°C and shows only approximately 1%

Table 1.8. Creep Rupture Properties of Molybdenum Alloys

Alloy	Irradiation		Test		Creep Rupture	
	Temp. (°C)	Fluence ^a $\times 10^{22}$ n/cm ²	Temp. (°C)	Stress $\times 10^3$ psi	Time (hr)	Elongation (%)
Molybdenum	--	0	750	24.18	87.1	50.5
	--	0	750	25.60	99.1	42.1
	--	0	750	27.02	57.0	37.7
	--	0	750	29.86	6.8	42.5
	800 ^e	6.1	750	25.6-33 ^b	4940 ^b	~ 1 ^b
	--	0	1200	6	124.5	66.8
	--	0	1200	8	13.7	54.8
	--	0	1200	17.2	0.05	65.5
	800 ^e	6.1	1200	8	427.6	43.1
	--	0	800	50	961 ^c	9.8 ^c
Mo-50% Re	--	0	800	60	21.7	22.1
	425	1.5	800	60-90 ^d	2119	1.1

^aFluence for neutrons $E > 0.1$ MeV.

^bTest not complete, stress is being increased in steps until failure occurs. At 33,000 psi for more than 400 hr.

^cTest terminated without failure.

^dLoad increased in steps. At 90,000 psi 210 hr before final failure.

^eDesign irradiation temperature 800°C. Operating irradiation temperature probably higher.

elongation. The stress has been gradually increased from an initial value of 25,600 psi to the present level of 33,000 psi during the test. This result is in marked contrast to results reported by Moteff et al.¹³ for creep tests on molybdenum at a stress of 18 kg/mm² (25,600 psi) at 750°C. For irradiation temperatures of 70,700 and 1000°C for neutron fluences of $\sim 1.4 \times 10^{20}$ neutrons/cm² (> 1.0 MeV) they found rupture lives that were 0.63, 2.8, and 4.0, respectively, times the rupture life of a control test. Creep rupture ductilities ranged from 0.75 to 1.0 times the control values. Our test at 750°C has been running for 50 times the life of the control specimen and has also had further stress increases without showing appreciable creep strain. The much greater strengthening observed in this sample than in Moteff's results may be a result of the higher fluence on the present specimen.

(d) Fractography of Tensile Specimens.- Typical tensile specimens were chosen for examination by scanning electron microscopy (SEM) to evaluate the effect of neutron irradiation on the fracture mode of molybdenum. All fracture surfaces examined were found to fall within two categories: (1) brittle fractures, characterized by mixed cleavage and intergranular separation surfaces, and (2) ductile fractures, with the entire fracture surface covered with the characteristic, equiaxed dimples of the normal rupture of a ductile material. None of the fractures examined showed mixed mode failures.

Typical areas on the fracture surfaces of the specimens that yielded the three tensile curves in Fig. 1.10 are shown in Fig. 1.13. In

¹³J. Moteff, R. C. Rau, and F. D. Kingsbury in Radiation Damage in Reactor Materials, vol. II, IAEA, Vienna (1969), pp. 269-281.

Fig. 1.13a, the irradiated Mo-0.5% Ti specimen which failed in a brittle manner, the fracture surface shows the characteristic cleavage failure pattern with 5 to 10% of the area showing grain boundary separation. The cleavage fracture features are similar to examples shown in the literature for bcc metals tested to failure below the DBTT. Figures 1.13b and 1.13c show the ductile fractures at 400°C in the irradiated specimen tested at 0.0002/min and the control sample tested at 0.02/min, respectively, and correspond to curves B and C in Fig. 1.10. Both are typical of ductile-mode failures and show only superficial differences.

Figure 1.14 shows samples of irradiated and control material tested at 650°C and Figs. 1.13b and 1.13c shows fractures at 400°C. The fracture surfaces of the irradiated and unirradiated samples show the dimples characteristic of ductile fracture. There do not appear to be any differences due to the neutron irradiation. It must be concluded from this examination of fracture surfaces that the neutron irradiation at 425°C has not affected the fracture mode of Mo-0.5% Ti in either the brittle or the ductile fracture range. This is in spite of the very large effect produced on the DBTT and the work hardening ability of the alloy.

Fracture surfaces of irradiated samples of Mo, Mo-0.5% Ti and Mo-50% Re that had been tested at 400°C were examined to determine the effects of alloying on the fracture mode. All three specimens had shown brittle behavior, failing with less than 0.2% elongation. Typical SEM fractographs of these samples are shown in Fig. 1.15, and show differences. The Mo-0.5% Ti, Fig. 1.15c, showed about 90% cleavage fracture surfaces and 5 to 10% grain boundary separation. The Mo sample, Fig. 1.15a,

Y1017 8

Y106244

100 μ

0.1mm

Fig. 1.14. Comparison of Ductile Fracture Surfaces in Irradiated and Control Samples of Mo-0.5% Ti. Irradiated sample on left. Both tests at 650°C and a strain rate of 0.02/min.

Y104560

Y104021

50μ

0.05mm

Y101697

Fig. 1.15. The Effect of Alloying on the Fracture Appearance of Irradiated Mo Alloys. (a) Unalloyed Mo irradiated to 3.45×10^{22} neutrons/cm². (b) Mo-50% Re irradiated to 1.5×10^{22} neutrons/cm². (c) Mo-0.5% Ti irradiated to 3.0×10^{22} neutrons/cm². All irradiations near 425°C. Tensile tests at 400°C at a strain rate of 0.02/min.

showed almost identical tensile properties but had a much greater fraction of grain boundary separation faces. It is estimated that in the Mo the fracture surface is approximately 50% cleavage faces and 50% grain boundary faces. The Mo-50% Re fracture surface is almost completely covered by cleavage faces, with no exposed grain boundary faces.

(e) Microstructure of Irradiated Samples.- The microstructure of Mo and Mo-0.5% Ti has been characterized in samples irradiated under conditions similar to the irradiation conditions for the tensile samples. A limited examination of specimens taken from tensile samples of irradiated Mo and Mo-0.5% Ti tensile samples show little difference between the two materials and little difference between those samples and the Mo sample described below.

The microstructure in a Mo sample irradiated to 2.5×10^{22} neutrons/cm² (> 0.1 MeV) at 425°C is shown in Fig. 1.16. The visible damage consists of barely resolvable dislocation loops and unresolved spots, which are assumed to also be loops. These loops form discontinuous linear arrays, with the strings of loops preferentially aligned along $\langle 110 \rangle$ directions. The loop density is 1.2×10^{17} cm⁻³ and the average diameter is estimated to be approximately 50 Å. There are no voids in the sample, and the observed strengthening is a result of the hardening produced by this very high loop concentration.

A segment of this same irradiated Mo sample was annealed for 1 hr at 900°C to investigate the thermal stability of the damage microstructure. A typical area in this specimen is shown in Fig. 1.17. The loop density has decreased by coalescence and growth to form a connected dislocation network and isolated, easily resolved loops. There is no evidence of

Fig. 1.16. The Microstructure Seen in Diffraction Contrast of a Mo Sample Irradiated to 2.5×10^{22} neutrons/cm² (> 0.1 MeV) at 425°C. The loop density is approximately 1.2×10^{17} cm⁻³ and the loop alignment is along $\langle 110 \rangle$ directions.

Fig. 1.17. The Microstructure of a Sample from the Same Specimen Shown in Fig. 1.16 Subjected to a Post-Irradiation Anneal of 1 hr at 900°C.

voids in this microstructure. Elen¹⁴ has demonstrated that voids formed in Mo at 440°C are stable under annealing to at least 800°C and upon annealing at 1200°C grow in size accompanied by a reduction in the void concentration. Our observation of the absence of voids in the annealed sample, coupled with Elen's demonstration of the void stability, confirms the absence of voids in the as-irradiated sample.

1.4 ✓ Studies of Swelling in Reactor Irradiated Metals

1.4.1 General

The importance of swelling of metal components to the reliable operation of a CTR has been established in recent reviews of irradiation damage problems expected during CTR operation.^{15,16} The swelling can interfere with dimensional tolerances, closing cooling channels or misaligning components, for example, or can impose buckling and bending stresses in components.

Swelling of metals is a result of cavity formation during irradiation. Cavities can form by the precipitation of insoluble gases in the metal, for example helium from (n,α) reactions, or by the precipitation of the vacancies that result from neutron displacement damage. Swelling in CTR components will result from the formation of cavities that are a hybrid between these two cavity types. It is expected that the cavities

¹⁴J. D. Elen in Voids Formed by Irradiation of Reactor Materials, BNES, Harwell (1971), pp. 51-62.

¹⁵F. W. Wiffen in Proc. Inst. Working Sessions on Fusion Reactor Technology, ORNL, June 28-July 2, 1971, CONF-710624.

¹⁶L. C. Ianniello, editor, Fusion Reactor First Wall Materials, WASH-1206, USAEC, Washington, D.C., (1972).

that form will not be true voids but rather will contain some helium at a pressure less than the bubble equilibrium pressure.

In this section we will summarize work on void formation and swelling of interest to the CTR program. The studies on vanadium were initiated at ORNL under the RDT Fuels and Materials Program. The rest of the specimens were irradiated by John Moteff while at GE-NSP. For convenience the results will be tabulated here and reviewed briefly. A major reservation that must be kept in mind in using these data is that the transmutation rates in the fission reactors are very much lower than expected in fusion reactors and that all swelling results may thus be modified.

1.4.2 Experimental

Results presented in this section were obtained on specimens that had been fully recrystallized prior to irradiation. The annealing treatments were appropriate for each material. The specimens of Nb, Mo, and Ta and alloys of each of these were machined from commercially supplied sheet stock. The V and V-20% Ti specimens were from laboratory fabricated materials. Purity levels in all materials were consistent with good commercial practice and are not representative of research grade metals. Irradiations were performed in the EBR-II reactor.

1.4.3 Results and Discussion

Void parameters that are of interest to the CTR program are tabulated in Table 1.9. Most of these data have been discussed previously.¹⁷ In these metals, as in all other metal systems examined,

¹⁷F. W. Wiffen, The Effect of Alloying and Purity on the Formation and Ordering of Voids in BCC Metals, ORNL-TM-3496 (1971); also in Radiation-Induced Voids in Metals, ed. by J. W. Corbett and L. C. Ianniello, AEC Symposium Ser. 26, CONF-710601 (April 1972), pp. 386-396.

Table 1.9. Void Parameters in Irradiated bcc Metals

Composition (wt %)	Irradiation Conditions		Void Parameters		
	Temp. (°C)	Fluence	Concentration	Average Diameter (Å)	Volume Fraction (%)
		$\times 10^{22}$ n/cm ^{2a}	$\times 10^{15}$ voids/cm ³		
V-I ^b	385	0.49	11.9	50 ± 10	0.08
V-II	385	0.57	118	45 ± 10	0.56
V-III	475	0.97	10.6	82	0.34
V-III	550	1.4	5.94	168	1.77
V-III	575	1.4	3.54	194	1.58
V-III	600	1.4	2.03	226	1.47
V-I	660	0.25 ^c	0	--	--
V-II	660	0.25 ^c	0 ^d	--	--
V-II	710	0.25 ^c	0 ^d	--	--
V-20% Ti	475	0.97	0	--	--
V-20% Ti	525	2.0	0	--	--
Nb	425	2.5	160	70	3.1
Nb	585	2.5	210	71	4.8
Nb	790	2.5	2.8	186	1.04
Nb-1% Zr	425	2.5	e	e	e
Nb-1% Zr	585	2.5	e	e	e
Nb-1% Zr	790	2.5	0.02-0.19 ^f	510	< 1.46 ^f
Mo	425	2.5	0	--	--
Mo	585	2.5	72	64	1.1
Mo	790	2.5	54	72	1.19
Mo-0.5% Ti	425	2.5	0	--	--
Mo-0.5% Ti	585	2.5	240	69	4.5
Mo-0.5% Ti	790	2.5	60	72	1.3
Ta	425	2.5	0	--	--
Ta	585	2.5	190	61	2.4
Ta	790	2.5	6.1	117	0.65
Ta	950-1050	4.4	≤ 0.3	130	≤ 0.03

^aFluence for E > 0.1 MeV.

^bVanadium starting interstitial content, I = 220 ppm, II = 1250 ppm, III = 1000 ppm.

^cORR irradiations.

^dHelium stabilized cavities formed surrounding boride precipitates. No cavities in matrix away from precipitates.

^eThese two samples probably void-free (conclusion tentative).

^fWide variation in void density.

the general features of void formation are the same. Within the temperature range over which voids form, the void concentration decreases and the void size increases as temperature increases and fluence is held constant. No bcc metals has been found to violate this general behavior. This effect of temperature on size and number of voids formed causes a temperature dependence of swelling that exhibits a peak intermediate in the temperature range of void formation. Two examples serve to illustrate this temperature dependence of swelling. All of the swelling data on neutron irradiated niobium that was available in 1971 was analyzed¹⁵ on the basis of linear extrapolation of fluence to define the swelling versus temperature relationship shown in Fig. 1.18. The data show a maximum in the swelling curve near 600°C and an upper limit on swelling at 900°C. The data for tantalum given in Table 1.9 very nearly bracket the temperature range for swelling. Data for tantalum are given in Table 1.9 and plotted in Fig. 1.19. There is a maximum in the swelling curve slightly below 600°C, but of course there are too few points to define accurately the swelling temperature relationship.

The first two entries in Table 1.9 show that the impurity content in vanadium influences void formation. This has been interpreted as showing that the interstitial impurities may stabilize the void nucleation process.¹⁷ Helium is effective in enhancing void nucleation in several fcc metals and alloys.¹⁸ In vanadium, irradiation to a low fluence at 600 and 710°C did not produce voids in the sample matrix. However, isolated boride particles were found in the lower purity vanadium and

¹⁸E. E. Bloom and J. O. Stiegler, J. Nucl. Mater. 36, 331 (1970).

Fig. 1.18. The Swelling of Niobium as a Function of the Irradiation Temperature. Data from several sources have been normalized to an intermediate fluence of 5×10^{21} neutrons/cm² (> 0.1 MeV) assuming that swelling is linearly dependent on fluence.

ORNL-DWG 71-11525

Fig. 1.19. The Swelling of Tantalum as a Function of Irradiation Temperature.

these were surrounded by a shell of cavities, as shown in the example in Fig. 1.20. This is clear evidence that helium, from the $^{10}\text{B}(n,\alpha)^7\text{Li}$ reaction, produces swelling far in excess of that found in helium-free material.

The effect of alloying on swelling has been investigated in only a few isolated cases. The cases for which we have data, in Table 1.9, show more than one effect. In Mo and Mo-0.5% Ti the void parameters are nearly identical after irradiation at 790°C but the alloy has a higher void concentration at 585°C, and thus more swelling. For vanadium alloys the effect is most dramatic; void formation is completely suppressed in V-20% Ti under irradiation conditions that produce swelling of 2% in V. Example microstructures in Fig. 1.21, for irradiations near 550°C, shows a high density of small voids in the V but only an irradiation-produced dislocation network in the void-free V-20% Ti. Void formation may be entirely suppressed in the alloy Nb-1% Zr for irradiation at 425 and 585°C. At 790°C voids form in both Nb and Nb-1% Zr but, as shown in Fig. 1.22, the void concentration is much reduced and the size much increased in the alloy. The best estimate is that the swelling is approximately equal in the Nb and Nb-1% Zr at 790°C.

The voids in some samples of Nb, Ta, Mo, and Mo-0.5% Ti are ordered. The ordering is not perfect; the voids seem to be displaced randomly small distances from the points of a bcc superlattice. The superlattice is parallel to the bcc metal lattice in all cases. An example of the void superlattice in a molybdenum sample is shown in Fig. 1.23 and the void lattice parameters for all cases in which the ordering was observed are given in Table 1.10. It has been suggested

Fig. 1.20. Microstructure of Vanadium Irradiated to 2.5×10^{21} neutrons/cm² (> 0.1 MeV) at 660°C in the ORR Reactor. No voids could be found in 99% of the area in the samples examined. The only cavities are those that are stabilized by the helium produced from $^{10}\text{B}(n,\alpha)^7\text{Li}$ reactions, such as those cavities shown surrounding the boride particle in the upper left of this photomicrograph.

Fig. 1.21. Microstructure of (a) Vanadium and (b) V-20% Ti Irradiated to 1.4×10^{22} neutrons/cm² (> 0.1 MeV) at 550°C. Void formation is completely suppressed in the alloy.

Fig. 1.22. Microstructures in (a) Niobium and (b) Nb-1% Zr
Irradiated to 2.5×10^{22} neutrons/cm² (> 0.1 MeV) at 790°C. Alloying
has reduced the void concentration and increased the void size.

Fig. 1.23. Void Ordering in Molybdenum Irradiated at 585°C.

Table 1.10. Summary of Void Ordering Data for
Neutron-Irradiated Refractory Metals^a

Composition (wt %)	Irradiation Temperature (°C)	Void Concentration (voids/cm ³)	Void Superlattice		
			Lattice Parameter (Å)	Density Points (cm ⁻³)	Super- lattice Occupancy
		x 10 ¹⁵		x 10 ¹⁵	
Mo	585	72	265	107	0.67
Mo-0.5% Ti	585	240 ^b	215	201	1.20
Mo	790	54	328	57	0.95
Mo-0.5% Ti	790	60	315	65	0.94
Nb	790	2.8	665	6.8	0.41
Ta	585	190	205	232	0.82

^aAll specimens irradiated to 2.5×10^{22} neutrons/cm² (> 0.1 MeV)

^bUpper limit.

that the ordered voids may represent a very stable defect configuration that signals the saturation of swelling.

1.4.4 Relationship of Swelling Results to CTR Operation

It is interesting to relate the available swelling results to the expected operating parameters specified in a conceptual fusion reactor design. One conceptual design with detailed temperature distributions is given by Mitchell and Hancox.¹⁹ The shape of a single cell that will form one segment of the blanket and vacuum wall is shown in Fig. 1.24, indicating the cooling scheme and the temperature and relative flux

¹⁹J.T.D. Mitchell and R. Hancox in 7th Intersociety Energy Conversion Engineering Conference, Am. Chem. Soc., Washington, D.C. (1972), pp. 1275-83.

ORNL DWG. NO 72-10897

Fig. 1.24. Schematic Design of a Single Cell that will form Part of the Blanket in the Mitchell and Hancox Conceptual Design (ref. 19). Coolant flow, coolant and wall temperatures, and relative neutron flux are plotted as a function of distance from the cell "nose".

distribution in the metal structure of the cell. If this cell were fabricated from niobium, the swelling could be related to the present results. Figure 1.25 shows microstructures that approximately relate to the service temperatures at the nose and base of the cell, although the neutron fluences are equal, not different by the factor of 5 expected in the reactor operation. The swelling values from Table 1.9 showed that the material at the temperature of the nose of the cell will have swelled approximately three times that at the cell base and the swelling maximum will actually occur somewhere on the cell side walls. This differential swelling on the various positions in the cell wall could cause buckling and interference between adjacent cells.

NIOBIUM WALL

Fig. 1.25. Comparison of Microstructures of Niobium Samples Irradiated at Two Temperatures Nearly Representative of Service Temperatures for the Blanket Cell shown in Fig. 1.24.

2. DAMAGE SIMULATION STUDIES

J. O. Stiegler

Neutron irradiation in conventional fission reactor test facilities cannot completely simulate the damage to be expected in a fusion reactor because of large differences in the neutron spectra. Since cross sections for transmutation reactions increase rapidly with increasing neutron energy, concentrations of hydrogen, helium, and solid transmutation products may be orders of magnitude higher in a CTR than in a fission reactor (see Table 2.1). Studies have shown that only a few atomic parts per million of helium injected into metals prior to irradiation in fission reactors can significantly increase the concentrations of voids and dislocation loops.^{1,2,3} Furthermore, solid impurities introduced at relatively low temperatures via transmutation reactions can result in the formation of precipitate particles⁴ or the introduction of non-equilibrium phases.⁵ In some cases transmutation produced precipitate particles may provide the principal source of radiation hardening.⁶

¹R. S. Nelson and D. J. Mazey, "Void Formation in Stainless Steel During Charged-Particle Irradiation at Elevated Temperatures," Radiation Damage in Reactor Materials, vol. II, Proc. Symposium, Vienna, June 2-6, 1969, IAEA, Vienna, 1969, p. 157.

²E. E. Bloom and J. O. Stiegler, J. Nucl. Mater. 36, 331 (1970).

³K. Farrell, A. Wolfenden, and R. T. King, Radiation Effects 8, 107 (1971).

⁴K. Farrell, J. O. Stiegler, and R. E. Gehlbach, Metallography 3, 275 (1970).

⁵M. C. Wittels, J. O. Stiegler, and F. A. Sherrill, Phys. Stat. Sol. 4, 533 (1964).

⁶K. Farrell and R. T. King, "Radiation-Induced Strengthening and Embrittlement in Aluminum," to be published in Met. Trans., 1973.

Table 2.1. Transmutation Rates^a

Metal	Reactor	Helium (ppm/yr)	Hydrogen (ppm/yr)	Solid Products (%/yr)
Nb	CTR wall ^b	270	890	1.4 (Zr,Mo,Y)
Nb	EBR-II core	4	180	--
V	CTR wall ^b	790	1500	~0.7 (Cr,Ti)
V	EBR-II core	3.5	100	--

^aD. Steiner, "The Nuclear Performance of Fission Reactor Blankets," Nucl. Appl. and Tech. 9, 83 (1970).

^bWall loading of 10 MW/m².

Since the neutron fluxes of currently available high energy neutron sources are too low to produce the amounts of radiation damage or transmutation products expected in a fusion reactor, we are attempting to simulate high energy neutron damage by heavy ion bombardment using a 6-MeV Van de Graaff accelerator. The displacement damage is produced in a metal specimen by bombardment with self ions; through the use of multiple ion sources, "transmutation products" are implanted in concentrations to be expected in a fusion reactor. Displacement rates produced by available currents of heavy ions may be as much as five orders of magnitude higher than in proposed reactor designs.⁷ As a consequence, a lifetime of damage can be introduced into a material in the matter of few hours, thus allowing the study of many variables and providing a quick turn around time for factoring new results into future experiments.

⁷G. L. Kulcinski, J. L. Brimhall, and H. E. Kissinger, "Production of Voids in Pure Metals by High-Energy Heavy-Ion Bombardment," Radiation-Induced Voids in Metals, Proc. 1971 Intern. Conf. Albany, New York, June 9-11, 1971, ed. J. W. Corbett and L. C. Ianniello, USAEC Office of Information Services, 1972, p. 449.

The simulation will not be exact, since differences exist in the primary knock-on spectra between bombardment by heavy ions, fission reactor neutrons and fusion reactor neutrons. At this time there is no evidence that for elevated temperature irradiations these differences will result in any difference in the form of the damage, but the degree of damage may differ. In stainless steel qualitatively similar damage structures have been produced by irradiation with fission reactor neutrons,⁸ 1-MeV electrons,⁹ 100-KeV¹ to 1.4-MeV protons,¹⁰ 12.3-MeV deuterons,¹¹ 20-MeV carbon ions,¹² 3 to 48-MeV nickel ions^{7,12} and 8-MeV tantalum ions.⁷ Differences in sizes and numbers of defects observed in these studies are believed to be kinetic in origin arising from differing damage rates occurring in the different irradiations. The size or shape of the displacement cascade or even the existence of a cascade appears to have little influence on the final form of the damage.

⁸C. Cawthorne and E. J. Fulton, "The Influence of Irradiation Temperature on the Defect Structures in Stainless Steel," The Nature of Small Defect Clusters, vol. 2, ed. M. J. Makin, Atomic Energy Research Establishment Report AERE-R-5269, Her Majesty's Stationery Office, London, 1966, p. 446.

⁹J. T. Buswell, "The Growth of Voids in Stainless Steel," Voids Formed by Irradiation of Reactor Materials, Proc. Conf. Reading, England, March 24-25, 1971, ed. S. F. Pugh, M. H. Loretto, and D.I.R. Norris, British Nuclear Energy Society, 1971, p. 283.

¹⁰D. W. Keefer, A. G. Pard, and D. Kramer, Radiation-Induced Voids in Metals, Proc. 1971 Intern. Conf. Albany, New York, June 9-11, 1971, ed. J. W. Corbett and L. C. Ianniello, USAEC Office of Information Services, 1972, p. 511.

¹¹J. S. Armijo and T. Lauritzen, Radiation-Induced Voids in Metals, Proc. 1971 Intern. Conf. Albany, June 9-11, 1971, ed. J. W. Corbett and L. C. Ianniello, USAEC Office of Information Services, 1972, p. 479.

¹²R. S. Nelson, J. A. Hudson, D. J. Mazey, G. P. Walters, and T. M. Williams, Radiation-Induced Voids in Metals, Proc. Intern. Conf. Albany, June 9-11, 1971, ed. J. W. Corbett and L. C. Ianniello, USAEC Office of Information Services, 1972, p. 430.

For these reasons we believe that ion bombardment provides a valid simulation of the damage produced by high energy neutrons. It is not without its drawbacks, however. Heavy ions have ranges of only a few microns in metals and along that range the damage is most intense in the final fraction of a micron. The damage consequently is introduced non-uniformly into only a small volume of material. This limits examination to electron microscopy techniques and prevents measurement of mechanical properties. For this reason neutron irradiation studies are a necessary complement to this program to provide guidance in estimating mechanical property trends and to give information on defect hardening mechanisms to allow ultimately the calculation of properties from microstructural observations.

There are problems also in relating changes produced by ion bombardment to those from neutron irradiation because of the accelerated rate at which the damage is introduced. This alters the relative importances of the different mechanisms by which point defects are annihilated and effectively shifts the temperature ranges in which various processes occur. A calculation indicates that a two order of magnitude increase in displacement rate will increase the temperature of peak swelling by about 100°C.¹³ There is no direct evidence on how changes in the displacement rate affect the shape of the swelling versus temperature curve or the maximum amount of swelling.

¹³R. Bullough and R. C. Perrin, "Theory of Void Formation and Growth in Irradiated Metals," Radiation-Induced Voids in Metals, Proc. 1971 Intern. Conf. Albany, June 9-11, 1971, ed. J. W. Corbett and L. C. Ianniello, USAEC Office of Information Services, 1972, p. 769.

The primary question is how should ion bombardment experiments be performed so that information relevant to fusion reactor operating conditions can be extracted from them. Although several programs have demonstrated the usefulness of ion bombardment in simulating neutron damage,^{7,12} none has examined the quantitative relationship between the two. This presents obvious difficulties not only in relating the two but also in comparing results from different ion bombardment techniques. The problem cannot be limited to a study of the characteristics of ion bombardment because quantitative studies of fission reactor induced swelling in pure metals are not available. Even in a material as thoroughly studied as stainless steel the empirical relationships between swelling, temperature, and fluence have been derived from a large number of isolated observations rather than from any systematic studies.¹⁴ In order to provide the baseline neutron irradiation data against which the ion bombardment results can be compared, we conducted during 1972 a controlled temperature reactor irradiation experiment on three pure metals, aluminum, nickel, and vanadium, to define fully the swelling and microstructural changes induced by neutron irradiation. In addition, we designed and constructed an ion bombardment chamber for use on ORNL's 6-MeV Van de Graaff and ORIC. We also conducted some preliminary experiments to measure the spatial distribution of gas atoms in metal foils using nuclear scattering techniques.

¹⁴H. R. Brager, J. L. Straalsund, J. J. Holmes, and J. F. Bates, "Irradiation Produced Defects in Austenitic Stainless Steel," Met. Trans. 2, 1893 (1971).

2.1 ~~The~~ Temperature Dependence of Fission-Reactor
Induced Swelling in Aluminum, Nickel, and Vanadium

K. Farrell

21,341

To provide a quantitative measure of the effect of irradiation temperature on swelling and microstructure, rods of high purity aluminum, nickel, and vanadium were irradiated in the B8 core lattice position in the Oak Ridge Research Reactor to fast neutron fluences ($E > 0.1$ MeV) in the range 0.5 to 1.0×10^{21} neutrons/cm². Temperature ranges were ~ 65 to 300°C , 300 to 750°C , and 350 to 900°C , for the aluminum, nickel, and vanadium, respectively. The experiment is described in detail in Section 2.2. An attempt was made to irradiate two specimens at each temperature to slightly different neutron fluences. This should allow us to extrapolate all results to a common fluence. Density measurements have been made to determine irradiation-induced swelling as a function of irradiation temperature. The measurements on aluminum show that swelling decreases with increasing temperature and does not occur above $\sim 185^\circ\text{C}$. This conclusion is confirmed by transmission electron microscopy of the specimens. Density measurements on the Ni and V were not so satisfactory because volume changes were small and because of interference from oxide films. Swelling data will be determined by transmission electron microscopy. Specimens have been prepared from all materials and are currently being examined. Full examination and evaluation of the experiment should be complete by mid-1973.

2.2 ORR-227; Temperature-Dependent Neutron-Damage Study

W. W. Davis

The object of this experiment was to irradiate in ORR, specimens of aluminum, nickel, vanadium, and alloys of molybdenum and niobium at controlled temperatures in the range from 200 to 900°C. Specimens were generally rods 1.0 in. long and nominally 1/8 in. diameter. The refractory metal specimens were encapsulated in stainless steel to prevent oxidation and contamination.

For temperatures of 600 to 900°C the specimens were suspended axially in cylindrical furnaces, four of which can be seen at right in Fig. 2.1. This arrangement depends on thermal radiation and gas convection to remove gamma heat.

To obtain specimen temperatures below 600°C additional cooling was necessary. These specimens were mounted in thick walled aluminum tubes which were welded to support arms and the arms were welded to a central water tube (see Fig. 2.1). The length and cross section area of support arms and the gas space between the specimen and its aluminum tube were designed to give the necessary cooling for each desired temperature. Electric heat for temperature control was provided by a heater winding fitted snugly over the tube. One or more chromel-alumel thermocouples were attached to each specimen.

The high purity aluminum specimens were in the form of a fluted fin. One long edge of the fin was welded to the central water tube and then a 3/8-in. aluminum tube was welded to the opposite long edge; its weight was supported by stainless steel pins to prevent deformation

Fig. 2.1. View of the Assembled Experiment for the ORR Irradiation Before Insertion into an Aluminum Can.

of the pure aluminum. A small spirally wound heater was inserted in the 3/8-in. tube, creating a temperature gradient between the heater and the cooling water tube through the aluminum fin. Thermocouples were attached at several places on the fin.

The experiment was enclosed in an aluminum can with dimensions of an ORR fuel element. The experimental assembly replaced a fuel element in position B8 in the ORR fuel lattice. Thermocouple leads, heater leads and a gas supply tube made up the control cable which extends through an access tube to the reactor pool surface. Here it was attached to an existing control system with a He-1% O gas supply, pressure controls, safety monitor, and 40 temperature controllers. The 1% O prevents formation of the more volatile lower oxides of chromium on the thermocouples.

When the reactor came to full power it was not possible to control the nickel or vanadium specimens at 200 or 250°C. By fitting control temperatures to gamma-heat temperatures we were able to cover the range of 300 to 900°C at 50°C intervals. This was possible since most specimens of a given metal were identical in size and metallurgical history. Except for the inability to achieve the low temperature (200 to 250°C) conditions, the irradiation experiment went well for the two operational cycles of the ORR - approximately 2000 hr at 30 MW.

2.3 The Ion Bombardment Chamber

21,343

A. Wolfenden

2.3.1 Design

The particle energies in our available accelerators are such that the penetration distances of the ions into the metals are of the order of

microns (Table 2.2). Thus the chamber must be constructed to hold relatively small specimens under controlled conditions of temperature (Table 2.3) and environment so that contamination of the bombarded materials can be avoided. To simulate transmutation reactions the 6-MeV Van de Graaff accelerator has been equipped with special multiple ion sources to provide us with 10 to 50 μ A of certain singly charged ions and a device to switch ion species rapidly. The accelerator and chamber will thus allow us to damage fusion reactor candidate materials with "self" ions and inject "transmutation" products in a controlled way to give a simulation of the damage expected.

The members of a team (Table 2.4) representing all facets of the project were involved in developing our design. The ion bombardment chamber and specimen assembly is shown schematically in Figs. 2.2 through 2.4.

The appropriate ion beams from the Van de Graaff enter the system through a valve assembly at the top. Their energies are reduced when necessary by the metal foils in the rotatable degrader wheel to give the chosen ion energies given in Table 2.2. Rotation of the degrader wheel is through a high vacuum rotary seal. This arrangement ensures that the radiation damage and the simulated transmutation products are deposited at the same depth in the specimen. So that a convenient specimen area can be irradiated and studied afterwards by transmission electron microscopy, it is necessary to sweep the beam in a circular manner. This sweeping is accomplished by the two sets of plates in the beam sweeping assembly. The required oscillating voltage on these plates is ~ 3 kV and the sweeping frequency must be about 3 kHz to ensure that the defects generated in

Table 2.2. Average Calculated Ranges of Ions in Selected Metals

Projectile Ion	Target Specimen	Ion Energy (MeV)	Projected Range (μm)
Al	Al	6.0	3.2
Si ^a	Al	6.4	3.2
Si ^a	Al	6.0	3.1
He	Al	1.0	3.2
H	Al	0.34	3.2
V	V	6.0	1.7
He	V	0.87	1.7
H	V	0.31	1.7
Ni ^b	Ni ^b	6.0	1.2
He	Ni	0.75	1.2
H	Ni	0.28	1.2
Nb	Nb	6.0	1.2
Zr ^a	Nb	5.9	1.2
He	Nb	0.66	1.2
H	Nb	0.25	1.2

^aSimulation of transmutation product.

^bFe into stainless steel should be similar to Ni.

Table 2.3. Melting Temperatures of Selected Metals and the Typical and Maximum Specimen Temperatures for Ion Bombardment Studies

Metal	Melting Temperature (T_m)* (°C)	Temperatures During Ion Bombardment	
		Typical (0.4 T_m) (°C)	Maximum (°C)
Al	660	100	294
Ni**	1453	417	690
V	1900	596	914
Nb	2468	823	1198

* T_m is the melting temperature in degrees Kelvin. Values have been converted to degrees Centigrade here for simplicity of comparison.

**Stainless steel is similar to Ni.

Table 2.4. Personnel Concerned with Irradiation Chamber for Ion Bombardment Studies of Metals and Alloys

J. A. Biggerstaff	Physics Division
W. W. Davis	Metals and Ceramics Division
B. C. Duggins	Instruments and Controls Division
K. Farrell	Metals and Ceramics Division
T. J. Golson	General Engineering Division
J. Googe	Instruments and Controls Division
J. T. Houston	Metals and Ceramics Division
J. W. Johnson	Physics Division
D. Kiplinger	Plant and Equipment Division
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Fig. 2.2. Schematic of the ORNL Ion Bombardment Chamber.

Fig. 2.3. Details of the Vacuum System of the ORNL Ion Bombardment Chamber.

Fig. 2.4. Schematic of the Specimen and Heater Assembly of the ORNL Ion Bombardment Chamber.

the specimen by the beam on one "visit" to a particular area have not vanished to sinks by the time of the next beam "visit". The specimen and its heater assembly are located at the bottom of the chamber (see Fig. 2.4 for details). The whole system is under high vacuum (Fig. 2.3) varying from about 10^{-9} torr at the specimen assembly to minimize specimen contamination, to 10^{-7} to 10^{-8} torr in the region of the sweep plates, to about 10^{-6} torr near the interface with the Van de Graaff machine. These variations in vacuum are controlled by the type of vacuum pump (electro-ion and oil diffusion pumps), liquid nitrogen baffles and water cooling coils, and strategically placed orifices to give differential pumping. A mechanical pump backs up the oil diffusion pump and also serves to "rough down" the whole ion bombardment chamber. Several ion gauges and vacuum valves are incorporated in the system. The whole chamber, with alignment devices, is mounted on a mobile frame.

Our specimen design avoids the problems encountered by the workers at Harwell and elsewhere who have used thin foils glued to specimen mounting plates. As shown in Fig. 2.4, our specimen material and specimen holder are one and the same thing — an integral unit situated within the controlled temperature zone. The temperatures at eight locations on the non-bombarded surface of the specimen will be monitored continuously. The auxiliary heater permits both precise specimen temperature control and beam calorimetry for flux and fluence measurements. Advantageous aspects of the specimen assembly are that all the heaters and the thermocouples are located out of the high vacuum and that different specimens may be inserted relatively easily via a single small flange.

2.3.2 Status of the Ion Bombardment Chamber on December 31, 1972

The design of the chamber is complete. Most of the machine-shop work on the body of the chamber is finished and the components are being leak-tested. The specimen assembly is being machined. The pumps and related vacuum equipment have been ordered; most items have been delivered. The instrumentation and controls for the whole system are in their final stage of design. Specimen preparation, both for the near-term and long-term experiments, is underway. The multiple-ion source on the Van de Graaff generator is operative. To date, beams of H, He, Al, and Fe (similar to Ni) ions at energies over 5 MeV have been achieved. Progress with Si, Ni, and V ions will take place soon after the alignment of a new analyzer magnet.

2.3.3 Initial Experiments to be Performed

That fast neutron damage can be simulated quickly using ion beams was demonstrated in 1969 by workers at Harwell.¹ Despite follow-up studies with a variety of ions,^{7,10,11,12} several major areas of uncertainty exist in our understanding of radiation damage produced in this way. Our early experiments will therefore be aimed at defining the interrelationships among displacement numbers, displacement rate, and types and distribution of damage and the relation of heavy-ion bombardment to neutron bombardment. As discussed above this study must come first if we are to get the full potential of this technique. We should then be in a position to extrapolate with confidence ion bombardment observations obtained relatively quickly to high-fluence neutron irradiation conditions in fusion reactors.

Our initial runs will be on specimens of high-purity aluminum. These studies, on identical material, will complement the neutron damage investigations described in a preceding section. The experiments will

provide the "calibration" for developing the relationships between neutron damage and ion damage. Specimens of high-purity nickel and vanadium will complete the series - Al, Ni, and V - receiving the "calibration" irradiations.

2, 3, 4 2.4 ✓ Some Effects of Neutronically Generated Gases in Metals

K. Farrell

The first wall of a fusion reactor will be subjected to temperatures in the range 600 to 1000°C, that is, about 0.3 to 0.5 of the absolute melting temperatures of the refractory metals Nb, Mo, and V which are currently being considered as candidate first-wall materials for a CTR. In this temperature range deterioration of mechanical properties and swelling and distortion from void formation can be expected, so there is clearly reason for concern over the effects of displacement damage. But experience with materials irradiated in fission reactors suggests that this problem might be alleviated by appropriate manipulation of alloying elements and microstructure to minimize void formation. A major problem area is likely to arise from the transmutation products. Solid transmutation products, if they are soluble, may be accommodated with little trouble or, if insoluble, may cause considerable hardening and embrittlement, as does transmutation-produced silicon in aluminum.¹⁵ However, the solid transmutation products may prove less worrisome than the neutronically generated gases.

¹⁵K. Farrell and R. T. King, "Radiation Induced Strengthening and Embrittlement in Aluminum," to be published in Met. Trans. (1973).

Table 2.1 showed that large quantities of helium and hydrogen are created by (n,α) and (n,p) reactions. An economic lifetime for the reactor might be 10 years during which the gases in a niobium first wall could amount to 2700 at. ppm helium and about 9000 at. ppm hydrogen at high power densities; a vanadium first wall would develop even higher quantities of gases. Hydrogen might diffuse out of the wall but the helium is likely to remain and can produce a number of highly undesirable effects. In the first place, as little as 0.1 at. ppm helium can reduce elevated temperature ductility in a creep test by as much as 50%.¹⁶ Second, the helium may cause swelling to occur earlier in the irradiation history of the wall.¹ Third, both helium and hydrogen may modify the size and concentration of voids^{3,2} to a stage where the voids become the major source of hardening and embrittlement in the host metal. Fourth, gas bubbles may develop, particularly on grain boundaries, at lower temperatures and neutron fluences than they occur in fission reactors. These bubbles could grow rapidly and link up to cause intergranular separation even in the absence of applied stresses. Some of these effects have recently been observed in aluminum containing large quantities of neutronic gases.

This aluminum was an alloy of high purity aluminum and about 2300 at. ppm Li in the form of an annealed rod about 2 in. long by 0.12 in. diameter. Irradiation in the strong thermal neutron environment of the HFIR at about 75°C ($0.37 T_m$) to fluences of 1.6×10^{21} neutrons/cm² ($E > 0.1$ MeV) and 3.7×10^{21} neutrons/cm² ($E < 0.414$ eV) converted more

¹⁶J. O. Stiegler and J. R. Weir, Jr., "Effects of Irradiation on Ductility," Ductility, Proc. ASM Seminar, October 1967, p. 311.

than 90% of the ^6Li to helium and the hydrogen isotope, tritium, creating about 2100 at. ppm of each. Moreover, since the lithium was completely soluble in the aluminum, the gases were generated homogeneously throughout the specimen. Some of the tritium may have diffused out but the helium is usually not mobile at 75°C . Note that these gas contents are a little less than the amounts of helium and hydrogen estimated for Nb and V in a 10-year period of irradiation in a fusion reactor at maximum power densities considered.

During hot cell disassembly of the capsule containing the Al-Li rod, the rod was accidentally allowed to fall about 1 in. onto a steel plate. It broke into several pieces in a completely brittle manner, the fracture surfaces being wholly intergranular. In marked contrast, an unirradiated rod was so weak and ductile that it could be easily bent through 360° using only the fingers of one hand! Hardness measurements gave 14.1 kg/mm^2 for the pure aluminum, 14.5 kg/mm^2 for the unirradiated Al- ^6Li alloy and a fivefold increase to 76 kg/mm^2 for the irradiated alloy. Transmission electron microscopy revealed the microstructure of the irradiated alloy to consist of a uniform distribution of dislocations and a high concentration of cavities, probably gas bubbles (Fig. 2.5). These bubbles ranged in size from about $100 \text{ \AA}$ diam to below the resolution limit of about $20 \text{ \AA}$; at least $5 \times 10^{16}/\text{cm}^3$ were visible. Bubbles were also present in relatively low concentrations on the grain boundaries but were several orders of magnitude larger than those in the matrix (Fig. 2.5b). The size and frequency of these large bubbles varied from one grain boundary to another, many being too large to contain within the $4000 \text{ \AA}$ thick foils. Immersion density measurements showed that irradiation had caused 0.5% swelling, versus 0.8% for pure aluminum irradiated

Fig. 2.5. Left, Dislocations and Cavities, Presumably Gas Bubbles in Irradiated Al-⁶Li Alloy. Right, large gas bubbles on a grain boundary. In this lower magnification picture the small gas bubbles in the matrix are barely visible.

under similar conditions. In the latter case, there were no bubbles on the grain boundaries but dislocations and cavities were present in the matrix, the cavities being typically about 500 Å diam with a concentration of $\sim 1 \times 10^{14}/\text{cm}^3$. The irradiated pure aluminum specimen remained ductile and had a hardness of 25 kg/mm², less than a twofold increase.

The irradiated Al-Li alloy could be snapped with very little force and no measurable ductility at room temperature. At liquid nitrogen temperature more force was required. Examination of the fracture surfaces showed that fracture occurred on those boundaries where considerable interconnection of large bubbles had occurred during irradiation (Fig. 2.6). Between 50 and 90% of the fracture area was already separated by cavitation prior to induced fracture. Fracture was completed by tearing and shearing of the small amount of intact grain boundary between the bubbles. It was observed, too, that tritium was continuously released from the irradiated Al-Li specimen at room temperature. Moreover, electron microscopy disks, on standing for about a year at room temperature, developed cracks on grain boundaries that had earlier shown no cracks. This might be caused by mobile tritium precipitating into the large grain boundary bubbles, building up pressure and expanding them.

Aluminum is not considered as a likely CTR first wall material. It was used here to demonstrate the effects of high gas contents; severe hardening and embrittlement of even the most ductile metals beyond the point of usefulness.

Fig. 2.6. Grain Boundary Fracture Surfaces in Irradiated Al-⁶Li Alloy, Showing Interconnected Bubbles and Tearing of Bubble-Free Regions. Broken at room temperature. Direct carbon replicas.

3. CHEMICAL COMPATIBILITY OF POTENTIAL BLANKET AND FIRST WALL MATERIALS

J. H. DeVan

We are assisting in the investigation of the compatibility problems associated with proposed fusion reactor design concepts. One major problem area concerns the environmental effect of the tritium breeding blanket on the plasma containment vessel. Other compatibility problems are associated with convective heat transfer systems used to derive energy from the reactor.

The high tritium breeding ratio and excellent heat transfer properties of metallic lithium makes it an attractive dual-purpose blanket-coolant material for CTR applications. We are conducting corrosion studies with lithium to elucidate problems that are peculiar to the fusion reactor concept.

3.1 Examination of the Forced-Circulation Lithium Loop

J. H. DeVan, B. Fleischer, R. L. Klueh, and E. L. Long, Jr.

From 1950 to 1970 we conducted corrosion studies of liquid lithium pursuant to its use as a high-performance heat-transfer fluid in advanced energy systems. Early studies centered on niobium-base alloys as container materials; however, to meet demands for higher thermodynamic efficiencies, the materials focus shifted to tantalum alloys, which allowed higher heat sink temperatures.

Our early corrosion studies with lithium^{1,2} were made in small closed loop systems in which we imposed a temperature gradient to induce flow by natural convection. These studies led to the construction and operation of an engineering-scale forced-circulation loop fabricated from the tantalum-base alloy T-111 (Ta-8% W-2% Hf). In June 1971, we began a metallurgical and chemical examination of the loop under contract with National Aeronautics and Space Administration-Lewis Research Center, and the examination was completed in 1972 as part of the Fusion Reactor Technology Program.

The principal value of the loop examination in terms of CTR interests is the comparison of high-velocity corrosion results, realized in the subject loop experiment, with the results obtained from earlier low velocity natural circulation loops. Since there is a close parallel in the behavior of T-111 and Nb-1% Zr in lithium, knowledge of the velocity effect with T-111 will allow us to realistically formulate the test requirements for future compatibility studies of niobium alloys. These loop results also greatly expand the limited body of test data that exist on the behavior of refractory metals in lithium under forced-flow conditions. Operating conditions for the subject loop are listed in Table 3.1.

The loop, shown schematically in Fig. 3.1, was fitted into a cylindrical vacuum enclosure 10 ft high by 4 ft diameter. The loop was

¹J. H. DeVan and C. E. Sessions, "Mass Transfer of Niobium-Base Alloys in Flowing Nonisothermal Lithium," Nucl. Appl. 3, 102-109 (1967).

²C.E. Sessions and J. H. DeVan, "Thermal Convection Loop Tests of Nb-1% Zr Alloy in Lithium at 1200 and 1300°C," Nucl. Appl. 9, 250-259 (1970).

Fig. 3.1. Forced Circulation Liquid Lithium Loop (FCLLL-1) Schematic.

Table 3.1. Loop Description

Temperature, °C	
Maximum	1370
Minimum	1205
Flow Rate, gpm	5.2
Maximum Velocity of Lithium, ft/sec	18
Maximum Reynolds Number	89,000
Total System Pressure Drop, psi	36
Resistance Heater Power, kW	30
Volume of Circulated Lithium, in. ³	300
Tubing Dimensions, in.	
Heater, Radiator,	
Outer Economizer Shell	7/8 OD x 0.065 in. wall
Inner Economizer Bore	1/2 OD x 0.065 in. wall

heated electrically and was cooled by direct radiation to the vacuum chamber wall. The loop temperature profile was accurately recorded by tungsten-rhenium thermocouples pot welded directly to the loop wall.

3.1.1 Test Results

After 3000 hr of operation, lithium was drained from the loop and the loop was flushed with liquid anhydrous ammonia to remove residual traces of lithium. The loop was then sectioned to retrieve insert specimens and to provide metallurgical and chemical samples of the loop wall.

The mass of container wall constituents transferred into and out of the lithium was determined by weighing 84 specimens which had been located at spaced intervals within the loop. The weight changes are

plotted in Fig. 3.2b as a function of loop position and are correlated against loop temperature (Fig. 3.2a). The maximum dissolution rate occurred at the point of maximum loop wall temperature and, in terms of uniform wall thinning, represents a surface loss of < 0.0001 in./year.

The specimens in the 1200°C pump inlet and outlet lines showed essentially constant weight gains of 0.6 to 0.7 mg/cm². The only other specimens to show weight gains were those just inside the economizer, where the weight gains decreased from about 0.7 mg/cm² at 1200°C to essentially zero at 1235°C.

The surfaces of the specimens that gained weight were covered with a crystalline deposit, and specimens that lost weight exhibited superficial grain-boundary grooving. These contrasting surface features, as revealed by scanning electron microscopy, are shown in Fig. 3.3. Although we have not yet identified the crystalline compound, electron microprobe analysis revealed that it contained up to 88% Hf. Chemical analysis of the specimens showed the concentrations of N, C, and O to be higher in the cooler regions of the loop (Fig. 3.2c). Hafnium surface concentrations, shown as a function of loop position in Fig. 3.2d, mirrored the weight change profile in Fig. 3.2b. Weight losses recorded for insert specimens in the dissolution zone of the loop corresponded closely to the calculated weight of hafnium and interstitial impurities lost from the same specimens. Thus there was little mass transport of tantalum, which is the major component of T-111.

Miniature sheet tensile samples were machined from a select number of the T-111 sheet specimens to evaluate changes in mechanical properties. Control tests were provided by sheet specimens which had

Fig. 3.2. Plots of Weight and Chemistry Changes Occurring as a Function of Specimen Position in Lithium-T-111 Forced Circulation Loop. (a) Loop temperature profile; (b) specimen weight change profile; (c) after-test concentrations of O, C, and Ni; (d) after-test concentration of hafnium at specimen surfaces.

Fig. 3.3. Surfaces of T-111 Sheet Specimens from the Forced Circulation Lithium Loop. (a) Maximum weight loss location; (b) maximum weight gain location; (c) higher magnification of b.

been annealed in two tungsten-mesh furnaces housed in the same vacuum enclosure as the loop. The furnaces operated for 1500 hr at 1093 and 1370°C, respectively, which corresponded to the lowest and highest temperatures around the loop circuit. We also evaluated sheet specimens in the before-test, i.e., as-received condition.

Mechanical properties of these specimens at room temperature are compared in Fig. 3.4. Both the control (vacuum-annealed) and lithium-exposed specimens decreased in ultimate and 0.2% offset yield strengths as compared to as-received material, while the percent elongation remained essentially unchanged. Specimens annealed at 1093 and 1370°C had similar tensile properties at room temperature. The specimens exposed to lithium at 1093°C had properties comparable to the annealed specimens, but lithium-exposed specimens at 1370°C were noticeably weaker than the annealed specimens. The loss in tensile properties at 1370°C reflects the removal of hafnium and nitrogen discussed above. We also noted that the grain size of the lithium-exposed specimens at 1370°C was considerably coarser than that of the control specimens annealed at 1370°C.

3.1.2 Significance of Results to CTR Program

It is informative to compare the present loop results with the results of a tantalum alloy thermal convection loop which circulated lithium for 3000 hr at a maximum temperature of 1350°C. The loop was constructed of the alloy T-222, which is similar in composition to T-111, as shown below.

Alloy	Nominal Composition, wt %			
	Hf	W	C	Ta
T-111	2	8	< 0.01	Bal
T-222	2.5	9.5	> 0.01	Bal

ORNL-DWG 72-9540R

Fig. 3.4. Effect of Operating Temperature on the Elongation, Ultimate Strength, and 0.2% Offset Yield Strength of the T-111 Miniature Tensile Specimens from the Forced Circulation Lithium Loop.

The loop was constructed of 1 in. OD $\times$ 0.065 in. wall tubing, and the lithium flow rate was approximately 7 ft/min.

Weight change results measured for insert specimens in the thermal convection loop test are compared against the loop temperature profile in Fig. 3.5a and b. Note the striking similarity in the magnitudes of the weight changes relative to the forced convection loop (Fig. 3.2b vs Fig. 3.5b). Also, like the forced convection loop, the weight change profile in the thermal convection loop correlated closely with the migration of hafnium and interstitial impurities, as determined from chemical analyses of the inserts and loop wall. The changes in surface concentration of hafnium around the loop are shown in Fig. 3.5c and are similar in magnitude and slope to those observed in the higher velocity test (Fig. 3.2d).

There appears to be almost no effect of flow rate on the mass transfer behavior of T-111 and T-222 in lithium. The major effect noted at both high and low flow rates involves the transport of hafnium and interstitial impurities from hotter to cooler regions of the loop. Tantalum transport remained negligible even at a velocity of 18 ft/sec and maximum loop temperature of 1350°C.

Hafnium transport in these systems shows an exact parallel with zirconium transport in Nb-1% Zr loop systems tested with lithium at 1200 and 1300°C. Note, for example, the similarity in the zirconium surface concentrations measured in Nb-1% Zr thermal convection loops,^{1,2} shown in Fig. 3.6, and the hafnium surface concentrations in Figs. 3.2b and 3.5c. Furthermore, the zirconium concentration changes, coupled with interstitial impurity migration, accounted for the bulk of the weight

Fig. 3.5. Plots of Weight and Chemistry Changes Occurring as a Function of Loop Position in Lithium-T-222 Thermal Convection Loop. (a) Loop temperature profile; (b) weight change profile; (c) after-test concentration of hafnium at specimen surfaces.

ORNL-DWG 67-11433

Fig. 3.6. Zirconium Surface Concentrations of Nb-1% Zr Loops Operated with Lithium.

changes occurring in Nb-1% Zr systems, and transport of niobium was almost negligible (in common with tantalum in the T-111 system). An earlier report² discussed the kinetics of zirconium transport in Nb-1% Zr lithium systems, and these same kinetic models appear to describe the transport of hafnium in our tantalum loops. The models also predict hafnium transport to be independent of the lithium velocity in accord with our loop results.

If one examines the present T-111 forced convection loop results and accepts that they have relevance to niobium-zirconium alloy systems, then the following conclusions can be drawn relative to corrosion program requirements for niobium alloys under the CTR technology program. We know from the results of Nb-1% Zr thermal convection loop tests that zirconium transport will constitute the principal corrosion effect in low-velocity lithium. The present loop results tell us that zirconium transfer will remain the principal effect at higher flow rates and can be effectively predicted from the results of low-velocity tests.

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